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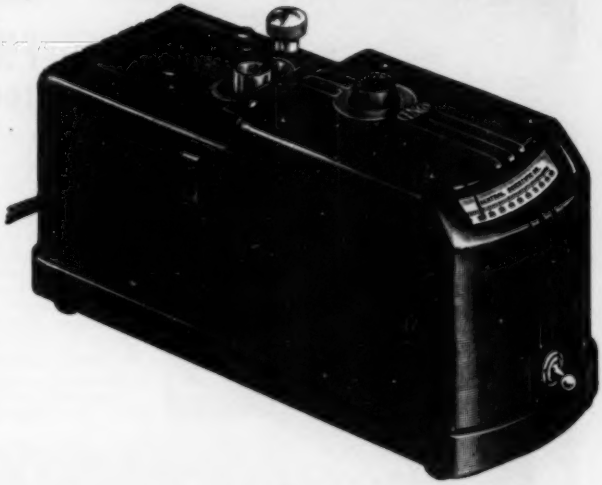

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CEREAL CHEMISTRY

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THE DEPENDENCE OF THE PHYSICAL AND CHEMICAL COMPOSITION OF THE CORN KERNEL ON SOIL FERTILITY AND CROPPING SYSTEM [1,2]

T. S. HAMILTON, BARBARA C. HAMILTON, B. CONNOR JOHNSON, AND H. H. MITCHELL

ABSTRACT

It has been found, in studies of forty samples of hybrid corn grown under different conditions of soil fertilization and crop rotation, that corn grown continuously year after year for almost three-quarters of a century on a soil type that is naturally productive, with no return to the soil of nutrients thus withdrawn, produced kernels about 26% smaller in size than the kernels of well-nourished corn. These runty kernels possessed the following physical and chemical properties as compared with normal corn, i.e., the same varieties of corn produced on other plots in the same year under the most favorable conditions.

The germ was small and accounted for some 17% less of the kernel than it should. Consequently the nutrients largely concentrated in the germ, the oil and the phosphorus, were all present in subnormal proportions. The endosperm was rich in starch but low in protein, because it was low in the flinty or horny portion of the endosperm carrying the more protein. In fact, all portions of the kernel were lower in protein than normal; in the entire kernel the protein deficit below normal was 30%. While the nicotinic acid content of the corn kernel had not been appreciably disturbed, the content of other vitamins that are concentrated in the germ may well have been.

The decrease in germ size and the lower oil content of the poorly nourished corn, in particular, have implications for the industrial processing of corn.

When these deficiencies in malnourished corn were corrected by proper soil and crop managment, a more wholesome food product resulted, although corn at its best is far from being a well-balanced diet for either man or animals. It is true that elevation of the protein content by these means resulted in a protein mixture which is poorer in nutritive quality than the low-protein ill-nourished variety, because of the greater prominence of zein with its marked deficiencies in lysine and tryptophane. But in animal feeding this may be no handicap, if corn can be supplemented with protein concentrates that are more than adequate in their content of these amino acids, and therefore markedly correct these deficiencies in corn proteins. This is particularly true of protein concentrates of animal origin, such as dairy and packing-house by-products.

Many investigations reported in the literature have shown that the composition of the corn kernel, particularly the protein and the

[1] Manuscript received October 23, 1950.
[2] Contribution from the Division of Animal Nutrition, University of Illinois, Urbana, Illinois.

oil content, can be greatly modified, and improved for various purposes, by appropriate breeding practices. The effect of soil fertilization on the corn crop has been studied mainly from the standpoint of yields. However, a few papers have been concerned with the relationship between soil fertilization and kernel composition (5, 10, 12). No papers have been found in the literature concerned with the effect of cropping systems on the composition of the corn kernel.

It is quite commonly supposed that the seeds of the grain crops, as compared with the vegetative portions, "are produced under a wide variety of environmental conditions with remarkable constancy of composition." Stubblefield and De Turk (10), from whom this quotation is taken, found that application of fertilizers to low-producing soil tends to increase the percentages of the respective nutrient elements in the grain until a complete treatment is applied. "Then, with well-balanced nutrition, and consequent greater growth, the percentages of all elements decline through dilution, with the exception of potassium."

The purpose of the experiments to be reported here was to determine whether, and to what extent, failure to maintain soil fertility by soil treatment and crop rotation will modify the chemical composition of the corn kernel and the proportions existing among its several discrete parts, germ, endosperm, hull, and tip cap. Does the penalty of poor yield associated with poor soil and poor crop management carry with it a further penalty of a seed crop of impaired value as food? The study of the effect of these unfavorable crop growing conditions on the physical make-up of the corn kernel was designed to aid in the elucidation of the chemical changes that might be detected in the whole kernel. It would also have a bearing on the value of such corn in commercial processing.

Materials and Methods

The samples of corn analyzed in this investigation were obtained from the 1946 harvest of the Morrow plots on the University Campus, which have been under continuous experimental control since 1876. They were secured through the courtesy of the Department of Agronomy after having been in storage for over a year.

The soil on which these plots are located is classified as predominately Flannagan silt loam. "It is naturally a productive soil and is representative of a vast area of prairie land in central and northern Illinois" (1). The surface of the plots is almost level with a slight depression from west to east, and the area is drained by a tile line adjacent to each plot.

The 1946 corn samples were grown on Plots 3 and 5. Plot 3 has been cropped continuously with corn for 74 years; since 1901, a corn, oats, red clover rotation has been practiced on Plot 5. In each plot, 5 corn hybrids (Ill. 246, Ill. 972, Ill. 21, Ill. 201 and U. S. 13) were grown and harvested separately, and samples from each hybrid in each plot were studied.

In 1904, each original plot was quartered into sub-plots equal in area to one-twentieth of an acre. The north two quarters of each plot were continued with no fertilizer treatment, while on the south two quarters a treatment practice consisting of manure, chiefly in amounts equal in weight to the air-dry weights of the crops removed in the respective plots, limestone, and phosphate (either bone or rock phosphate) was established. To the date of planting of the 1946 crop, a total of 8.85 tons of limestone and 129 to 144 tons of manure have been applied to the south quarter plots. In addition, 6.6 tons of rock phosphate have been applied to the southwest quarters and 1.65 tons of bone phosphate to the southeast quarters. Limestone was last applied for the crop of 1943, but no phosphate has been applied since 1925.

Eight quarter-plots with five hybrids to each quarter plot furnished forty samples of corn for study. For each sample, the weight of 100 kernels was determined in triplicate, and chemical samples representing the whole kernel were analyzed for moisture (in partial vacuum), total nitrogen (using mercury as a catalyst), petroleum ether extract and total phosphorus by approved methods of the Association of Official Agricultural Chemists. Phytic acid phosphorus was determined by the method of Earley (2), and nicotinic acid by the method described by Johnson (7). The nitrogen in these samples was fractionated into a fraction soluble in 0.5 *M* sodium sulfate, a fraction in 71% ethanol, and the residual nitrogen. Tryptophane and lysine were determined microbiologically on a few of the samples by the method of Henderson and Snell (3).

The physical composition of the corn kernels grown under different soil conditions and under different cropping systems was determined by hand dissection of 100 kernels of each sample into tip cap, germ, hull, floury endosperm and horny endosperm after soaking the kernels in water. The different fractions were then dried in partial vacuum, weighed, and samples pooled from the different hybrids in each experimental plot were analyzed, in so far as available material permitted, for the same constituents as were the whole kernels, and by the same methods.

Stubblefield and De Turk (10) have observed that "Weather conditions exert a pronounced effect, with the result that crops grown on

the same plot in succeeding years may vary as much in chemical composition as crops grown in different plots." The weather data for Urbana tabulated by Page (8) show that the mean monthly temperatures during the growing season of 1946 were very similar to the average monthly temperatures for the period of recorded data (1889–1946). The mean monthly precipitations for 1946 were higher than the average for 57 years for May and June, and lower for July, August and September. For the period April 1 to September 30, the total precipitation was 20.07 in. for 1946 and 20.85 in. for the period 1889 to 1946.

The data were analyzed statistically by the paired-comparison method of Student (11), in order to reveal the effects of (a) fertilization, with four plot comparisons, (b) crop rotation, with four plot comparisons, (c) fertilization and crop rotation combined, with two plot comparisons, and (d) the type of phosphate fertilizer used, i.e., bone or rock phosphate, with two plot comparisons. Paired differences within each hybrid and between pairs of plots were pooled to give 19 degrees of freedom for (a) and (b), and 9 degrees of freedom for (c) and (d). The actual plots compared for these four purposes are indicated in the footnotes to the tables containing the statistical analyses of the average data summarized in corresponding tables. Most of these paired comparisons compare east plots with east plots, and west plots with west plots, thus avoiding complications that may be associated with the larger yields of corn on the east plots.

Results

Chemical Composition of the Corn Kernels. The average results of the chemical analyses of the whole corn kernels in the eight experimental plots are given in Table I. The statistical analyses will be found in Table II. Table I also contains the yield of corn in the different plots for the 1946 season. It will be noted that the east plot yields were always higher than the corresponding west plot yields. From Tables I and II the following conclusions may be drawn concerning the effect of soil treatment, crop rotation and type of phosphorus fertilizer on the chemical composition of the corn kernel:

(1) The fertilization applied to the soil in these plots definitely increased the weight of kernel, and the content of protein, fat, and total phosphorus, but depressed slightly the nicotinic acid content. It increased the percentage of the total phosphorus present as phytic acid and the percentage of total nitrogen soluble in alcohol (zein nitrogen), while depressing the percentage of the total nitrogen insoluble in alcohol and in sodium sulfate solution (mainly glutelin

TABLE I

THE AVERAGE CHEMICAL COMPOSITION OF THE WHOLE CORN KERNELS FROM THE DIFFERENT EXPERIMENTAL PLOTS WITH THE TOTAL YIELD PER PLOT

Plot	Treatment and Crop Rotation[1]	Yield	Average Dry Wt. of 100 Kernels	Chemical Composition of the Whole Kernel on the Dry Matter Basis					Fractionation of Nitrogen in % of Total Nitrogen		
				Protein, N×6.25	Ether Extract	Total P	Phytin P as % of Total	Nicotinic Acid	Soluble in 71% Alcohol	Soluble in 0.5 *M* Na_2SO_4	Residual N
		bu./acre	*g.*	%	%	%		*µg./g.*	%	%	%
3NE	No treatment Continuous corn	30.3	22.91	7.5	4.3	0.23	75.7	28.6	22.1	23.6	54.1
3NW	No treatment Continuous corn	16.0	22.25	7.1	4.5	0.24	79.6	31.5	21.8	25.7	52.4
3SE	LMnr. bP Continuous corn	87.7	27.66	8.6	4.6	0.27	85.3	29.6	30.3	23.5	46.2
3SW	LMnr. rP Continuous corn	81.6	27.75	8.5	3.5	0.27	81.1	25.4	27.0	22.6	50.3
5NE	No treatment Corn, oats, r. clover	83.1	27.22	9.6	4.6	0.21	74.3	29.9	26.1	21.5	52.5
5NW	No treatment Corn, oats, r. clover	68.5	24.77	9.7	4.5	0.20	72.1	29.7	26.5	19.2	54.4
5SE	LMnr. bP Corn, oats, r. clover	123.3	31.38	10.3	4.9	0.28	81.2	29.0	32.2	21.6	46.2
5SW	LMnr. rP Corn, oats, r. clover	119.3	29.61	10.4	4.7	0.30	78.2	27.0	36.6	21.5	41.8

[1] L = limestone; Mnr = manure; bP = bone phosphate; rP = rock phosphate.

TABLE II

THE STATISTICAL ANALYSIS OF THE DIFFERENCES BETWEEN THE AVERAGE PLOT VALUES GIVEN IN TABLE I

	Average Dry Wt. of 100 Kernels	Chemical Composition of the Whole Kernel on the Dry Matter Basis					Fractionation of Nitrogen, in % of Total Nitrogen		
		Protein, N ×6.25	Ether Extract	Total P	Phytin P as % of Total	Nicotinic Acid	Soluble in 71% Alcohol	Soluble in 0.5 *M* Na_2SO_4	Residual N
	g.	%	%	%		μg./g.	%	%	%
Effect of soil treatment									
No. of plot comparisons[1]	4	4	4	4	4	4	4	4	4
Mean difference	4.82	0.96	0.21	0.06	5.98	−2.14	7.41	−0.21	−7.23
t value	19.90	9.56	2.80	9.18	3.57	2.33	8.12	−0.38	−6.70
Effect of crop rotation									
No. of plot comparisons[2]	4	4	4[6]	4	4	4	4	4	4
Mean difference	3.10	2.09	0.06	−0.006	−3.96	0.12	5.06	−2.91	−2.03
t value	8.93	16.67	0.95	0.95	−3.05	0.17	5.10	−4.79	−1.85
Effect of soil treatment plus crop rotation									
No. of plot comparisons[3]	2	2	2	2	2	2	2	2	2
Mean difference	7.92	3.06	0.27	0.05	2.02	−2.02	12.47	−3.12	−9.26
t value	19.39	17.15	3.84	8.46	1.01	1.73	10.08	−4.32	−10.89
Effect of kind of phosphorus fertilizer									
No. of plot comparisons[4]	2[5]	2	2	2[7]	2	2	2	2	2
Mean difference	0.84	−0.04	0.29	−0.01	3.63	2.75	−0.61	0.52	−0.17
t value	1.82	0.44	2.66	2.08	2.59	2.71	−0.39	1.09	−0.10

[1] 3SE-3NE; 3SW-3NW; 5SE-5NE; 5SW-5NW; with 19 degrees of freedom. For P = 0.05, t must equal 1.73; for P = 0.02, t must equal 2.20.
[2] 5SE-3SE; 5SW-3SW; 5NE-3NE; 5NW-3NW; with 19 degrees of freedom.
[3] 5SE-3NE; 5SW-3NW; with 9 degrees of freedom. For P = 0.05, t must equal 1.83; for P = 0.02, t must equal 2.40.
[4] 3SE-3SW; 5SE-5SW; with 9 degrees of freedom.
[5] For 5SE-5SW, t = 3.40, P = 0.014.
[6] For 5NW-3NW, t = 4.30, P = 0.006.
[7] For 5SE-5SW, t = 6.8, P = 0.002.

nitrogen), without appreciably modifying the proportion of nitrogen soluble in sodium sulfate solution (mainly albumins and globulins).

(2) The cropping system practiced on these plots contributed to an increased kernel size and particularly to an increased protein content. It definitely depressed the percentage of the total phosphorus in the form of phytic acid without exerting any appreciable effect on the contents of total phosphorus, fat, or nicotinic acid. It contributed to the increase in the proportion of the total nitrogen as zein, while definitely depressing the proportion soluble in 0.5 *M* sodium sulfate solution (albumins, globulins and non-protein nitrogen). It probably also depressed the proportion of residual nitrogen, mainly glutelin nitrogen.

(3) The combination of fertilization and crop rotation significantly increased the weight of kernel by an average of 80 mg., the protein content by about 3 percentage units, the fat content by almost 0.3% and the phosphorus content by 0.054%, without significantly disturbing the proportion of the phosphorus present as phytic acid or the content of nicotinic acid. In the experience of Hunt, Rodriguez and Bethke (6), also, it is difficult to disturb the nicotinic acid content of corn by altering the soil conditions, or even by hybridization. The proportion of the total nitrogen as zein was markedly increased by 12.5%, and those proportions as albumins and globulins and as insoluble forms, mainly glutelins, were decreased by 3.1 and 9.3 percentage units, respectively.

(4) Bone phosphate fertilization, applied to the east plots, is associated with a higher content of fat and of nicotinic acid in the corn kernel and with a higher proportion of phosphorus as phytic acid than is rock phosphate fertilization, applied to the west plots, with indications also of a lower content of phosphorus and a slightly heavier weight of kernel associated with bone phosphate applications. No appreciable associations of the fractionation of the nitrogen of corn and the type of phosphorus application to the soil is revealed by the data of Table II. However, the interpretations of these associations is complicated by the consistently larger corn yields of the east plots, indicative of appreciable differences in soil fertility between east and west plots.

Tryptophane determinations were carried out on samples of all five hybrids for Plots 3NE, continuous corn with no soil treatment, 5SW, corn in rotation with oats and red clover with limestone, manure and rock phosphate fertilization. The average amount of tryptophane per g. of corn was 416 micrograms and 543 micrograms, respectively, equivalent to 0.63 and 0.59 g. per 16 g. of nitrogen in each case. The

differences between averages were highly significant, since for all hybrids the same relation held.

The lysine analyses were completed only for hybrids Ill. 21 and Ill. 201 in Plots 3NE and 3NW, respectively, and hybrid Ill. 201 on plot 5SW. The results on the first plots were 1.87 and 2.50 mg. per g. of corn, and on the third plot 2.26 mg. per g. of corn, equivalent, respectively, to 2.88, 3.96, and 2.36 g. of lysine per 16 g. of nitrogen.

The results expressed to 16 g. of nitrogen testify to the higher proportion of zein in the high-protein corn grown with soil treatment and crop rotation, since zein is deficient, or lacking, in tryptophane and lysine. The results expressed in weight of amino acid per g. of corn give the advantage to the high-protein corn in spite of its higher proportion of zein in the combined proteins.

Physical Composition of the Corn Kernels. The dissection of 100 kernels of corn from each of the 40 samples (5 hybrids from each of 8 plots) was undertaken in order to explain more fully the differences in chemical composition that may be revealed by the chemical analysis of the whole kernels. A second purpose was to secure information of the effects produced by differential soil and crop treatment on the value of the corn in commercial processing as that value may depend upon the proportions of germ, hull, endosperm and tip cap.

TABLE III

THE AVERAGE PHYSICAL COMPOSITION OF THE CORN KERNELS FROM THE DIFFERENT EXPERIMENTAL PLOTS WITH THE TOTAL YIELD PER PLOT

Plot	Treatment and Crop Rotation[1]	Yield	Dissected Parts in Per Cent of Whole Kernel on the Dry Matter Basis				
			Hulls	Tip Caps	Germs	Floury Endosperm	Horny Endosperm
		bu./acre					
3NE	No treatment Continuous corn	30.3	5.0	1.2	8.0	52.8	32.9
3NW	No treatment Continuous corn	16.0	4.9	1.3	9.0	59.8	25.2
3SE	LMnr. bP Continuous corn	87.7	5.0	1.0	9.3	40.2	44.4
3SW	LMnr. rP Continuous corn	81.6	5.2	1.0	10.0	39.0	44.9
5NE	No treatment Corn, oats, r. clover	83.1	4.9	1.0	9.3	42.2	42.6
5NW	No treatment Corn, oats, r. clover	68.5	4.6	0.8	9.4	42.7	42.4
5SE	LMnr. bP Corn, oats, r. clover	123.3	4.7	0.8	9.7	34.2	50.6
5SW	LMnr. rP Corn, oats, r. clover	119.3	4.9	0.7	10.7	34.1	45.6

[1] L = limestone; Mnr = manure; bP = bone phosphate; rP = rock phosphate.

TABLE IV

THE STATISTICAL ANALYSIS OF THE DIFFERENCES BETWEEN AVERAGE PLOT VALUES GIVEN IN TABLE III

	Dissected Parts in % of Whole Kernel				
	Hulls	Tip Caps	Germs	Floury Endosperms	Horny Endosperms
Effect of soil treatment					
No. of plot comparisons[1]	4	4	4	4	4
Mean difference	0.08	−0.18	1.02	−12.5	11.6
t value	0.20	−2.21	5.53	−7.42	7.00
Effect of crop rotation					
No. of plot comparisons[2]	4	4	4	4	4
Mean difference	−0.24	−0.28	0.67	−9.7	9.5
t value	−2.95	−3.99	4.44	−6.27	6.21
Effect of soil treatment plus crop rotation					
No. of plot comparisons[3]	2	2	2	2	2
Mean difference	−0.18	−0.46	1.69	−22.2	21.0
t value	−1.15	−2.20	4.13	−9.29	8.80
Effect of kind of phosphorus fertilizer					
No. of plot comparisons[4]	2	2	2	2	2
Mean difference	−0.20	0.07	−0.81	0.65	0.28
t value	−1.27	1.40	2.22	0.46	0.13

[1] 3SE-3NE; 3SW-3NW; 5SE-5NE; 5SW-5NW; with 19 degrees of freedom. With P = 0.05, t must equal 1.73; for P = 0.02, t = 2.20.
[2] 5SE-3SE; 5SW-3SW; 5NE-3NE; 5NW-3NW; with 19 degrees of freedom.
[3] 5SE-3NE; 5SW-3NW; with 9 degrees of freedom. For P = 0.05, t must equal 1.83; for P = 0.02, t must equal 2.40.
[4] 3SE-3SW; 5SE-5SW; with 9 degrees of freedom.

The average results of these operations for each of the experimental plots are summarized in Table III; the statistical analyses of average plot differences are given in Table IV.

In what might be called normal kernels of corn, i.e., those secured from plots 5SE and 5SW, the hulls compose about 4.8% of the whole kernel, the tip cap about 0.8%, the germ about 10.2%, the floury endosperm about 34.1%, and the horny endosperm about 50.1%.

The statistical analysis of differences between fertilized and unfertilized plots, continuous corn and corn in rotation with oats and red clover, and bone phosphate and rock phosphate, justifies the following conclusions:

(1) The soil treatment employed increased the proportion of germ and of horny endosperm, while it decreased the proportion of tip cap, and floury endosperm, while exerting no appreciable effect on the proportion of hull.

(2) The crop rotation employed exerted the same effects on the physical composition of the corn kernel as soil treatment, but generally to a somewhat lesser degree. In addition it definitely depressed the proportion of hull.

(3) As might be expected, the effects of a combination of soil treatment and crop rotation were additive, resulting in an average increase of 1.7 percentage units in the proportion of germs, an average increase of 21.0 percentage units in the proportion of horny endosperm, an average decrease of 22.2% in the proportion of floury endosperm, and an average decrease of 0.46 percentage units in the proportion of tip cap. The effect of the combined factors on the proportion of hull was indeterminate.

(4) The type of phosphate fertilizer employed in the southeast and southwest plots exerted no marked effect on the physical composition of the corn kernel, and no highly significant differences between averages for the bone phosphate and rock phosphate treated plots were observed. Rock phosphate fertilization was associated with a higher proportion of germ ($P=0.028$) than was bone phosphate fertilization, by an average of about 0.8 percentage units.

Chemical Composition of Physical Fractions of Corn Kernels. The chemical composition of the physical fractions of the corn kernels from the different experimental plots was necessarily determined in pooled samples, because of the small amounts available for analysis from individual hybrids. The results of these analyses are collected in Table V. Certain conclusions are obvious from the table, although statistical analysis of average plot differences is precluded.

The protein (nitrogen) content of all parts of the kernel was consistently higher in the plots on which a crop rotation was practiced than in the plots supporting a continuous corn crop. Fertilization alone seemed to exert no influence on the protein content of the germ, but probably increased slightly the protein content of the endosperm.

The oil content of the different parts of the kernel seemed to be independent of soil treatment and cropping system.

In all 4 plot comparisons of the effect of phosphate fertilization on the phosphorus content of the germ, an average increase was observed, while in the 4 plot comparisons of the effect of crop rotation on the phosphorus content of the germ, an average decrease was observed. The same comparisons with reference to the other parts of the kernel revealed similar and consistent relations between fertilized and non-fertilized plots. A depressing effect of crop rotation on the phosphorus content of the endosperm fractions was not clear-cut. Crop rotation, but not soil treatment, was consistently associated with a change in the average percentage of phytin phosphorus on total phosphorus, the change being an increase.

The nicotinic acid content of the different parts of the corn kernel tended to be depressed by the application of fertilizer, but the association is not perfect and the average differences not great.

TABLE V

THE AVERAGE PERCENTAGE COMPOSITION OF THE DIFFERENT FRACTIONS OF THE CORN KERNEL DETERMINED IN POOLED SAMPLES FROM THE DIFFERENT PLOTS AND EXPRESSED ON DRY MATTER BASIS

Plot	Soil Treatment and Crop Rotation	Anatomical Part	Protein (N ×6.25)	Ether Extract	Total P	Phytin P in % of Total	Nicotinic Acid	N Fractionation in % of Total N in Anatomical Part		
								Soluble in 0.5 *M* Na_2SO_4	Soluble in 71% Ethanol	Residual N
							μg./g.			
3NE and 3NW	No treatment Continuous corn	Germs	17.3	34.5	1.82	87.9	29.5	52.3	2.5	45.2
		Hulls			0.03	37.0	11.5			
		Floury endosperms	6.2	1.6	0.08	54.9	31.1	13.2	26.1	60.8
		Horny endosperms	6.8	0.6	0.02	21.7	22.7	7.3	45.1	47.6
3NW	No treatment Continuous corn	Germs	16.8	36.1	2.10	75.1	25.9			
		Hulls			0.02	26.3	10.1			
		Floury endosperms	6.2	1.1	0.06	43.0	28.2			
		Horny endosperms	7.2	0.8	0.04	37.1	20.5			
3SE	LMnr. bP Continuous corn	Germs	18.0	35.3	2.09	85.3	32.3			
		Hulls					12.6			
		Floury endosperms	7.4	1.7	0.09	57.8	23.9	11.2	23.5	65.3
		Horny endosperms	8.0	0.6	0.03	20.7	21.2	6.1	43.9	50.0

TABLE V—*Continued*

Plot	Soil Treatment and Crop Rotation	Anatomical Part	Protein (N ×6.25)	Ether Extract	Total P	Phytin P in % of Total	Nicotinic Acid	N Fractionation in % of Total N in Anatomical Part		
								Soluble in 0.5 *M* Na_2SO_4	Soluble in 71% Ethanol	Residual N
							μg./g.			
3SW	LMn. rP Continuous	Germs	17.0	33.9	2.13	83.5	23.6			
		Hulls			0.03	65.6	16.8			
		Floury endosperms	6.7	1.2	0.06	50.8	20.7	8.8	28.3	63.0
		Horny endosperms	7.7	0.8	0.04	12.2	27.2	6.5	49.2	44.3
5NE	No treatment Corn, oats, red clover	Germs	18.4	35.2	1.57	88.2	30.0			
		Hulls					12.7			
		Floury endosperms	8.4	1.2	0.05	48.9	25.9	8.7	26.6	64.7
		Horny endosperms	8.8	0.6	0.03	20.0	25.0	5.3	46.5	48.2
5NW	No treatment Corn, oats, red clover	Germs	18.0	34.2	1.47	82.2	28.6	60.5	4.1	35.4
		Hulls					10.2			
		Floury endosperms	8.3	1.2	0.05	60.8	27.6	11.0	30.6	58.4
		Horny endosperms	9.0	0.6	0.03	14.8	28.3	6.1	51.0	42.9
5SE	LMnr. bP Corn, oats, red clover	Germs	18.8	35.8	1.94	87.0	22.1			
		Hulls			0.02	47.4	10.2			
		Floury endosperms	8.9	1.5	0.08	56.3	24.3	8.7		
		Horny endosperms	9.6	0.6	0.03	23.3	23.7	5.4	48.5	46.1
5SW	LMnr. rP Corn, oats, red clover	Germs	18.4	32.9	2.06	86.3	27.0	58.8	4.5	36.7
		Hulls			0.03		11.9			
		Floury endosperms	9.1	1.1	0.07	59.7	23.5	9.1	38.2	52.7
		Horny endosperms	10.1	0.6	0.03	10.3	24.2	5.6	58.3	36.1

The data concerning the fractionation of the protein in the different parts of the kernels and in different experimental plots are so fragmentary as to render interpretation hazardous. The horny endosperm in all plots contained a much greater proportion of its nitrogen in the form of zein than the floury endosperm. The germ contains only a trace of zein; in a specially prepared sample of corn germ, 69.5% of its nitrogen was soluble in 0.5 *M* sodium sulfate solution (albumins, globulins, proteoses, etc.), 2.9% was soluble in 71% alcohol (zein), and 27.5% (probably mainly glutelin) was insoluble in both solvents.

The even distribution of nicotinic acid throughout the kernel, excepting the hull, is noteworthy. For all samples, the average contents of this vitamin, in micrograms per g. of dry matter, were 27.4 for the germ, 25.6 for the floury endosperm, 24.1 for the horny endosperm, and 12.0 for the hull.

Considering the corn samples of plots 5SE and 5SW, that were grown under the most favorable conditions, as representing most nearly adequately nourished kernels, the germ, constituting 10.2% of the kernel, contained 75% of the total phosphorus, 82% of the phytin phosphorus, 78% of the oil, 19.4% of the nitrogen, but only 9.6% of the nicotinic acid. Rogozínski (9) was unable to detect any phytic acid phosphorus in any fraction of the corn kernel but the germ.

The observations recorded on the changes in the physical composition of the corn kernel with changes in plot treatment throw considerable light on the chemical changes observed in the whole kernel. The increased protein content of the kernel associated with soil treatment and crop rotation is brought about by an increase in the proportion of those parts of the kernel containing the higher protein content, i.e., the germ and the horny endosperm, together with an increase in the protein content of the germ, but particularly the endosperm. The change in the character of the total protein of the kernel with soil treatment and crop rotation, viz., a greatly increased proportion of zein (alcohol-soluble protein) and a corresponding decrease in the proportion of the other fractions, may be traced to the greatly increased proportion of horny endosperm, the most important carrier of zein. The finding that increasing the protein content of corn by modifying growing conditions brings about a greatly increased proportion of horny endosperm in the kernel, readily detectable by naked-eye examination of a longitudinal section, is in harmony with the early observation of Hopkins, Smith and East (4) working with corn samples containing different levels of protein induced by selective breeding.

The increased proportion of germ in the better nourished corn

kernels accounts for their somewhat higher fat content and phosphorus content.

The slight, if any, effect of soil treatment and cropping system on the nicotinic acid content of the corn kernel is a result of the even distribution of this vitamin throughout all parts of the kernel except the hull.

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SOME CHARACTERISTICS OF BARLEY, MALT AND WORT GUMS[1]

W. O. S. MEREDITH, E. J. BASS, and J. A. ANDERSON

ABSTRACT

Gum-like polysaccharides were isolated from barley, malt and wort. The wort polysaccharide is the main viscous principle in wort. Acid hydrolysis followed by filter-paper chromatography showed that the principal sugars in all gums were glucose, arabinose and xylose, but galacturonic acid, galactose and mannose were also present in trace amounts. The gums are therefore related to the arabo-xylo-glucosans and galacto-mannans. The raw gums contain 3 to 3.5% of nitrogen which is completely removed by alkali treatment. Raw malt and wort gums were higher than raw barley gum in pentose content and in relative amounts of arabinose. All alkali-treated gums were higher than corresponding raw gums in pentoses and the relative amount of xylose was higher than that of arabinose. The malt gum may be derived from the barley gum by loss of a component, principally glucose. The relations between the various gums are discussed and the existence in the barley of several arabo-xylo-glucosans of different composition as mixtures or agglomerates is suggested.

The presence in barley and malt of complex polysaccharides other than starch, which are related to the plant gums, has been recognized for many years. The early work on barley gums or amylans was undertaken by O'Sullivan, Lintner, and Brown and is reviewed by Hind (6, p. 70). More recently, Piratzky and Wiecha (16) isolated a gummy material from barley and short-grown malt that was highly viscous in aqueous solution. As this gum was not present in well grown malts, they considered that it was characteristic of under modified malts. Analysis of the gum by the methods then available showed that glucose was the only sugar present. Recent developments in carbohydrate chemistry and techniques, particularly the application of chromatographic procedures, have facilitated the study of cereal gums. Preece, Ashworth, and Hunter (17) have recently presented information on barley and malt gums, and Perlin (15) has dealt with wheat gum. Additional investigations of barley, malt and wort gums are presented herein.

These studies are the result of searches for the viscous principle in malt worts. The investigations of Meredith and Sallans (11,12) on malts made from different varieties indicated that high wort viscosity

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was associated with low wort nitrogen content. This suggested that worts with low wort nitrogen content contained higher proportions of high molecular weight material than those with higher nitrogen content. However, as wort viscosity appeared to reflect extent of general malt modification—low viscosity being characteristic of well modified malts—the source of the high molecular weight material could not be definitely established. A later study (9) of the effects of mashing temperature on wort nitrogen content and viscosity indicated that the latter was dependent on components other than nitrogen. These studies were continued and the main viscous principle in wort was isolated (10). This material contained nitrogen, gave a negative test for starch by the iodine test and positive phloroglucinol tests for pentosans; it thus appeared to be a polysaccharide. The isolation procedures for recovery of the polysaccharide from wort have now been applied to barley. Chromatographic separations of the sugar components of the gums from barley, malt, and wort are reported, together with analytical data on the ratios of glucose to pentoses.

Materials

A sample of Montcalm barley grown at the University of Manitoba in 1949 was used. Some of this barley was malted in this laboratory by the standard procedures and the small samples were bulked and thoroughly mixed before aliquots were withdrawn for use. The barley and malt were stored in airtight containers in order to minimize changes in characteristics. The barley contained 6.7% moisture and 2.05% nitrogen. The analysis of the malt was: moisture, 4.4%; extract, 75.2%; saccharifying activity, 100°L.; wort nitrogen content, 1.0% of wort solids.

Isolation of Gums

Raw Gums. Barley was ground in a Wiley mill with a 1 mm. sieve to provide a whole barley meal. Endosperm flour was obtained by milling the barley, after tempering to 14% moisture, in an Allis-Chalmers mill to a short patent flour in approximately 40% yield. The meal or flour was extracted at 20°C. for 2 hours with distilled water. The ratio of flour to water was 1 to 8, and the mash was stirred continuously during the extraction process. The mash was centrifuged and the supernatant liquor, which was slightly hazy, was poured into 3 volumes ethyl alcohol with stirring. A stringy precipitate settled within an hour and was recovered by centrifuging. The gum was dried by solvent exchange using ethyl alcohol, acetone, and ethyl ether, in that order. The final product was then air-dried.

Malt gum was obtained by extracting ground malt (Miag mill fine grind) at 70°C. with distilled water, in a ratio of 1 part malt to 8 parts

water, for 2 hours. The mash was then cooled and filtered, and the liquor was poured into 3 volumes alcohol with stirring. The gum was recovered in the same way as barley gum. A temperature of 70°C. rather than 20°C. was selected to increase the yield of gum and to inhibit enzyme action; at higher temperature greater yields can be obtained but filtering of the mash becomes increasingly difficult. While "malt gum" has been used to designate the preparation obtained in this way, the reader will appreciate that it might well have been called a "70°-wort gum."

Wort gum was prepared from wort obtained by the Congress mashing procedure normally used for the determination of malt extract (2). This involves a final malt-water ratio of 1:8, and mashing, with continuous stirring, is carried out by holding at 45°C. for 30 minutes, then raising the temperature to 70°C. at the rate of 1 degree per minute. The mash is maintanied at 70°C. for 1 hour, cooled and filtered. The wort was treated as for barley extract to recover the gum.

Although the final period of extraction of malt in preparation of wort gum is carried out at 70°C., at which temperature many enzymes in malt are known to be inactivated, the early stage of mashing at 45°C. to 70°C. permits extensive enzymatic degradation of malt. As a result, wort prepared by the Congress procedure differs markedly in certain properties and in composition from wort prepared by mashing directly at 70°C. It is of particular interest in the present studies that the latter wort is more viscous than the former.

Alkali-Treated Gums. The raw gums contained nitrogen, which was removed by refluxing a 1% solution of gum in normal sodium hydroxide for 2 hours in a boiling water bath.

Alkali-treated gums were readily recovered from the alkaline digests of raw barley and endosperm gums. The liquor was cooled and insoluble material was removed by centrifuging. The supernatant liquor was poured into three volumes alcohol and a precipitate was recovered in the usual way by centrifuging and solvent exchange.

The raw malt and wort gums did not produce a precipitate when the cooled alkaline digest was poured into alcohol. However, a precipitate which was recovered in the usual way, was obtained when the alkaline digest was acidified with hydrochloric acid before pouring into alcohol. The alkaline solutions were not centrifuged before they were acidified.

Fractionation. Fractionation of the raw gums obtained by alcoholic precipitation, by extraction with hot and cold water or by precipitation with varying ratios of alcohol afforded fractions that were essentially identical as far as viscosity measurements and qualitative chromatographic analysis were concerned. The only procedure that produced materials that differed from the raw gums was alkali digestion

This indicated some differences between the barley and malt gums and also some similarities.

Properties of Gums

Methods. Two per cent solutions were prepared by mixing gum and distilled water and stirring the mixture gently till the gum was completely dispersed. The liquid was then centrifuged to remove small amounts of insoluble material. Viscosity measurements were made in an Ostwald viscosimeter at 30°C. on solutions of maximum concentration and these were diluted stepwise with water and further measurements made. The data were then plotted as viscosity-concentration curves to provide comparisons between gums at equivalent concentrations.

TABLE I

ANALYTICAL DATA ON GUMS

Sample	Yield (Dry Basis)	Nitrogen	Viscosity of 0.5% Aqueous Solution
	%	%	cp.
Whole barley meal			
Raw gum	3.1	3.52	1.35
Alkali-treated gum	31.1[1]	0	1.42
Endosperm			
Raw gum	3.2	3.55	3.05
Alkali-treated gum	59.0[1]	0	1.69
Malt			
Raw gum	3.0	2.92	1.30
Alkali-treated gum	32.0[1]	0	1.64
Wort			
Raw gum	3.8	3.30	1.22
Alkali-treated gum	46.0[1]	0	1.70

[1] Yield as per cent of raw gum.

Solutions of raw barley and endosperm gum were unstable, and viscosity decreased considerably during a 24 hour period from the time the solutions were prepared. Despite this instability, results were reproducible to about 0.02 centipoises when viscosity measurements were made at standardized intervals from time of preparation.

Nitrogen determinations were made on the gums by the A. A. C. C. procedure (1) recommended for flour.

All gums gave negative tests for starch when tested with iodine solution.

Results. Data on yield, nitrogen content and viscosity of solutions of the various gums are given in Table I. The yields of raw gum from all sources are similar and reasonably high, around 3%, and there are no great differences in nitrogen content.

The solubility of the raw barley gum was quite low; concentrations

of above 0.5% were not readily obtained. The other raw gums were much more soluble, and concentrations of slightly above 1% were obtained. The alkali treated gums were soluble to the extent of about 2%.

The instability in solution of the raw barley and endosperm gums may be attributed at least in part to enzymatic cleavage of the molecules by enzymes precipitated with the gum. Some other factors also influence stability of these gums in solution as different methods of preparing solutions produce widely different viscosities. The malt and wort gums and all the alkali-treated gums were stable in solution.

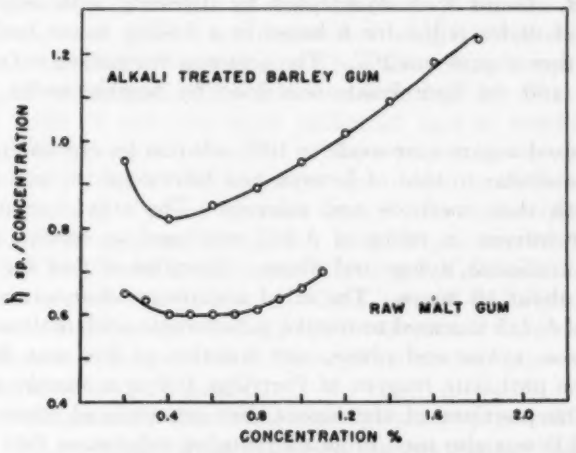

FIG. 1. Viscosity/concentration vs. concentration curves for gums.

The viscosities listed in Table I are for 0.5% solutions of the gums. The raw endosperm gum was more viscous than the raw barley gum, but the raw malt and wort gums were somewhat lower than the barley gum in viscosity. All the alkali-treated gums, with the exception of the endosperm gum, were more viscous than the raw gums. Alkali treatment appears to have concentrated the viscous principle, but this does not rule out the possibility that at least some of the nitrogen in the raw gums may contribute to viscosity as part of a protein-carbohydrate complex.

The viscosities of the solutions at various concentrations provided additional information on the characteristics of the gums. When intrinsic viscosity was plotted against concentration, the curves for all gums showed minima at about 0.4% to 0.6% concentration. The curves for alkali-treated barley gum and raw malt gum, which are typical of the series, are shown in Fig. 1. The increase in intrinsic

viscosity at dilute concentrations is typical of polyelectrolyte systems (4) which suggests that the gums are polyelectrolytes. This evidence of polar groups in the gums is not conclusive for the presence of specific constituents, but suggests that complex groups exist in the gums. Polar groups in the raw gums may be attributed to the presence of protein, but polar groups in the alkali-treated gums, which are nitrogen-free, may arise from polar sugar derivatives, somewhat similar to pectic-acid, that are known to be present in some plant gums.

Sugar Components of Gums

Method. Gums were hydrolysed by digesting with normal sulphuric acid under reflux for 6 hours in a boiling water bath. The concentration of gum was 2%. The acid was neutralized with barium carbonate and the hydrolysate was dried by heating under reduced pressure.

The mixed sugars were made to 10% solution for chromatography. Apparatus similar to that of Jermyn and Isherwood (8) was used together with their methods and solvents. The ethyl acetate-acetic acid-water solvent in ratios of 3:1:3 was used to resolve maltose, mannose, arabinose, xylose and ribose. Duration of flow for this solvent was about 40 hours. The ethyl acetate-pyridine-water solvent in ratios of 5:2:5 was used to resolve galacturonic acid, maltose, galactose, glucose, xylose and ribose, and duration of flow was 20 hours. The aniline phthalate reagent of Partridge (14) was mainly used for locating the positions of the sugars, but ammoniacal silver nitrate solution (13) was also used to locate reducing substances that did not react strongly with the other reagent.

Results. The qualitative chromatographic analysis of the sugar components showed that at least eight sugars are present in each of the gums. Glucose, arabinose and xylose are the principal sugars, but galacturonic acid, galactose and mannose are also present in small amounts. Ribose is definitely present in the alkali-treated barley and endosperm gums and is also present in trace amounts in the other gums. This sugar is not regarded as a characteristic component of plant gums and may be derived from a nucleo-protein such as was found in wort by Hopkins (7). A material that is equivalent to maltose in Rg value[2] in both solvents is also present in trace amounts in all gums. Another material with Rg intermediate between those of galacturonic acid and maltose with the ethyl acetate-pyridine-water solvent is also present. Actually, these slower moving spots may represent oligo-saccharides that arise from incomplete hydrolysis of the gums.

[2] Rg is ratio of distance the spot travelled from base line to distance travelled by glucose spot.

Quantitative Study of Sugars

Method. The ferricyanide procedure customarily used in cereal laboratories for measurement of saccharifying activity (1) was modified to measure smaller amounts of sugar. The method differs but little from the original Hagedorn-Jensen procedure (5) but it is more flexible.

The standard solutions were 0.05 *N* alkaline potassium ferricyanide and 0.05 *N* sodium thiosulphate. These were diluted to from 0.025 *N* to 0.0025 *N* for use in analysis, but the acetic acid-salt solution suitable for 0.05 *N* reagents was used irrespective of the strength of the other solutious. The concentrations of ferricyanide and thiosulphate were varied according to amount of sugar present. When the amounts of pentoses were low, glucose was determined using 0.025 *N* solutions and pentoses with 0.0025 *N* solutions. When the pentoses were present in higher concentrations 0.005 *N* solutions were used for all sugars.

The 0.025 *N* solutions were calibrated against solutions of D-glucose, L-arabinose and D-xylose. The relations beween amounts of sugar and net volume of thiosulphate were linear for each sugar, but a different factor is required for each sugar. The sugar equivalents of 1 ml. 0.0025 *N* sodium thiosulphate were: D-glucose, 0.083 mg.; D-xylose, 0.087 mg.; and L-arabinose, 0.100 mg. This value for glucose equivalent is close to the value of 3.2 mg. per ml. 0.10 *N* thiosulphate (1) and indicates that the relation between amount of sugar and amount of thiosulphate is independent of thiosulphate concentration when using otherwise identical procedures.

Chromatograms for quantitative analyses were prepared by streaking the 10% solutions of the mixed sugars in a narrow band ($\frac{3}{16}$ in.) across the width (9 in.) of the paper; three streaks were usually superimposed. After removal from the chamber, the papers were air-dried and a 1½ in. strip was cut from each lengthwise edge of the paper. These marker strips were developed and the positions of the sugars on the remaining section were determined. The paper was then cut into strips corresponding to the sugars. These 6 in. sections were then cut in half and used for quantitative determination of the sugars.

The sections of filter paper containing the individual sugars were folded and placed in a 25 ml. test tube to which was added 10 ml. ferricyanide solution. The tube was covered and placed in a boiling water bath for 20 minutes and then cooled. The contents of the tube were rinsed into a 125 ml. Erlenmeyer flask with 3 volumes distilled water, and 25 ml. acetic acid salt solution and 1 ml. starch-iodide solution were added. The flask contents were then titrated with sodium thiosulphate solution until the blue color was discharged. A blank determination was made on a piece of sugar-free filter paper cut

from the chromatogram. The net titration, blank minus sugar titration, was converted into amount of sugar by means of reference graphs.

Direct boiling of the filter paper gives high blank values and these are variable, but consistent results for sugar determinations have been obtained by determining blanks on sections of paper cut from areas close to each sugar spot. An elution procedure like that of Dent's (3) might be preferable, but useful information was obtained by the direct boiling procedure.

Results. Quantitative measurements for glucose, arabinose and xylose were made on papers that had been irrigated with the ethyl acetate-acetic acid-water solvent. This solvent did not resolve galacturonic acid and galactose from glucose, so that the section of the paper used for determination of glucose contained small amounts of these two materials. Further, it is possible that the amounts of these would vary from gum to gum. However, the results of the analyses provide information on the relative amounts of the two pentoses and the relations between those and the total amount of glucose, galacturonic acid and galactose, calculated as glucose. The results of these determinations, as ratios of the pentoses to glucose (uncorrected for the other two sugars) are given in Table II.

TABLE II

RATIOS OF PENTOSES TO GLUCOSE IN GUMS

Sugar	Raw Gum				Alkali-Treated Gum			
	Barley	Endosperm	Malt	Wort	Barley	Endosperm	Malt	Wort
Glucose[1]	1	1	1	1	1	1	1	1
Arabinose	.10	.11	.23	.18	.46	.44	1.20	1.14
Xylose	.09	.09	.13	.14	.58	.35	1.42	1.23

[1] Probably containing traces of galactose and galacturonic acid.

The raw barley and endosperm gums are similar in composition; the pentose content is quite low, and arabinose and xylose are present in roughly equal amounts. The raw malt and wort gums are also similar in composition, and are higher than the raw barley gums in pentose content. Further, the amounts of arabinose in the malt and wort gums are greater than those of xylose.

A very different picture is presented by the data on alkali-treated gums. The pentose contents are much higher than those of the corresponding raw gums, and xylose appears in greater amounts than arabinose. The differences in pentose content between the malt and wort gums on one hand, and the barley gums on the other hand, is maintained

and even accentuated, but other differences are shown. The raw malt and wort gums contained higher relative amounts of arabinose than the raw barley gums, but all the alkali-treated gums, except that from endosperm, contain more xylose than arabinose. These differences in amount and distribution of pentoses can thus give rise to considerable speculation not only on the constitution of the gums but also on the relations between them.

Discussion

The object of these studies was to determine whether the barley and malt gums differed, and whether the malt gum was derived from the barley gum. The available data on yield, viscosity, nitrogen content, and sugar components of the various gums provided some information bearing on these points. Limited comparisons may also be made with the results obtained by Preece *et al.* (17) from a similar study. Their results are also useful when considering certain hypotheses regarding relations between the gums.

The yields and nitrogen contents of the raw gums are all similar, so that these measurements failed to differentiate the gums. There were some differences between the gums in solubility, but these add little to the discussion. The difference between raw barley and raw endosperm gum in viscosity in 0.5% solution is quite marked. A reasonable explanation is that enzymes that cause instability of gum are present in greater concentration in the whole barley grist than in the endosperm flour, so that the endosperm gum is not attacked during extraction to the same extent as the whole barley gum. The barley gum must contain a high proportion of the endosperm gum; both gums contain the same relative amounts of sugar but may differ in molecular size. Similarly, the malt and wort gums are related, but the wort gum has been subjected to more degradation during preparation than the malt gum and the former is lower in viscosity. This is in accord with the viscosities of the liquors from which the gums were prepared. The Congress wort had a viscosity of 1.45 centipoises and the liquor obtained by mashing at 70°C. to prepare malt gum had a viscosity of 1.65 centipoises. The ratios of the principal sugars in malt and wort gum are quite similar. Recognition of the similarities between the barley and endosperm gums and between the malt and wort gums simplifies the picture and the main comparisons may be made between the barley and malt gums.

Qualitative analyses of the sugar components of the raw barley and malt gums suggest that they are related. The principal sugars are glucose, arabinose and xylose, but galactose, galacturonic acid and mannose are also present. Quantitative analyses for the principal

sugars show some differences between the gums. The malt gum is higher than the barley gum in pentose content and contains more arabinose than xylose. The barley gum contains equal amounts of arabinose and xylose.

Barley and malt gums are both hydrolysed readily by 0.1 *N* sulphuric acid, which suggest that a high proportion of the sugar residues are in the furanose ring form. Enzymatic studies, which will be reported in a later paper, have shown that all the gums are attacked by an enzyme system that reduces viscosity and increases reducing power. The products of hydrolysis have not been identified, but when this is accomplished a useful key to the relations between the gums may well be provided.

At this point it is advisable to consider the results of Preece *et al.* (17). Our yields of gum are considerably higher than the total of the two gums isolated by Preece *et al.* from both barley and malt, but the viscosities of our gums in 0.5% aqueous solution are of the same order as those of their gums. Preece *et al.* did not report nitrogen contents for their materials. Their malt gums were higher in pentose content than barley gums isolated in the same way and, although quantitative measurements of the individual pentoses were not given, Preece *et al.* reported that in three of the four gums xylose content was greater than arabinose content. Our results thus agree with those of Preece *et al.* in the difference between the malt gum and barley gum in pentose content, but they differ in the relative amounts of arabinose and xylose. However, it is important to note that Preece *et al.* were able to obtain two components from each of their gums and that alkali was used in their isolation procedures. These facts assist in the interpretation of our results.

Alkali treatment of our barley and malt gums revealed that they differed in certain respects and were similar in others. Barley gum can be precipitated from alkaline solution by addition of alcohol, but alkaline solutions of malt gum have to be acidified before a precipitate is produced by addition of alcohol. However, not all the gum originally soluble in alkali is precipitated, which suggests that the gums contain two or more components. Further, the alkali-treated products from all gums are considerably higher in pentoses than the corresponding raw gums and also contain more xylose than arabinose. Alkali treatment therefore produced information that can be used to formulate a hypothesis regarding the relation between the gums.

A series of decreasing glucose content is shown by the sequence raw barley, raw malt, alkali-treated barley and alkali-treated malt gums. The ratio of arabinose to xylose also changes, but both alkali-treated gums are higher in xylose than arabinose. These results suggest that

raw barley gum is a mixture of polysaccharides that are similar in many respects, but differ in relative amounts of the three principal sugars. Malting of barley produces a change in relative glucose and xylose contents of the gum, and alkali treatment removes glucose and arabinose preferentially.

These changes in composition may be interpreted in various ways. The gums may be mixtures of a glucosan, an araban and a xylan, which may be removed to different degrees by the processes used in the studies Alternatively, sugars may be present as gluco-arabans, gluco-xylans, or arabo-xylans. However, as all three sugars are present in all gums, and in all fractions obtained from the gums, it seems most probable that the three sugars are combined as arabo-xylo-glucosans, and that at least three of these complexes occur in the raw barley gum. One that is principally glucose but also relatively higher in xylose than arabinose may be partly removed by malting, and one relatively higher in arabinose may be partly removed by alkali treatment. Barley gum would contain all three complexes. Malt gum would contain more of one pair and alkali-treated barley gum would contain more of a different pair. Alkali-treated malt gum would consist mainly of one complex, that which is common to all gums. The nitrogen complexes of the raw gums and the possible galacto-mannan do not enter into this scheme, but they do not affect this line of reasoning. The difference between barley and malt gum in precipitability in alkaline solutions is not accounted for, but there may be interactions between the different polysaccharides that cause co-precipitations.

Preece *et al.* (17) agree that the gums are probably mixtures of polysaccharides, but believe that the malt product is derived from barley hemicelluloses that are made soluble during malting. It seems more probable to us that at least some of the malt gum is derived from the barley gum. Further studies, directed especially to fractionation of products isolated in various ways, are obviously required to elucidate the complete picture.

Acknowledgment

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A SMALL SCALE DOUGH EXPANSION TEST FOR THE ESTIMATION OF WHEAT QUALITY [1,2]

H. MILLER, J. EDGAR, and A. G. O. WHITESIDE

ABSTRACT

A small scale dough expansion test to evaluate bread making properties of wheat is described. In this test the expansion of a fermenting wheat flour dough in a weak solution is measured on a calibrated tube. A formula for making up the dough containing monoammonium phosphate, calcium acid phosphate, salt, sugar, and yeast is given.

The results with two series of flours show that the expansion figure is related to loaf volume and may provide information over and above that supplied by protein content alone. This is found to be true where high protein is not associated with large loaf volume. Such a test may be useful in indicating bread making qualities where only small samples of wheat are available, such as in wheat breeding projects.

The degree and rate of expansion of fermenting doughs has been used in various forms for over 50 years as an index of flour quality.

Hays and Boss (1) used an expansion test under the name of "The Bakers' Sponge Test" and found it valuable "in aiding to throw out

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[2] Published as contribution No. 153 of the Cereal Division, Dominion Experimental Farm, Ottawa, Canada and as paper No. 286 of the Associate Committee on Grain Research (Canada).

the less desirable varieties of wheats." Maurizio (2) mentions a modification of this test using tubes instead of jars, and reducing the amount of flour to 30 g. Chidlow did much to popularize the test on a large scale but this has been superseded by the small scale baking test.

The original expansion tests were all conducted in air. Saunders (3) introduced a modification by fermenting doughs in water, in which the estimation of flour quality was taken from the length of time a dough ball would endure fermentation in water before disintegration began. Cutler and Worzella (4) from their work recommended the use of wheat meal instead of flour in this method, though they admit that no correlation existed between dough ball disintegration time and loaf volume. This has been found by the present authors to be particularly true for Canadian hard red spring wheats. Pelshenke (5) used it in his small scale test work and found it "extremely necessary with this method to make four determinations to get dependable results."

The purpose of this paper is to describe a modification of the dough ball expansion test which was devised particularly to distinguish quality in Canadian wheat varieties. It is essentially a small scale test, and should therefore be of value to the wheat breeder in working with early generations.

Procedure for Making a Dough Expansion Test

To obtain the potential expansion of a dough as little support and restriction as possible should be given it. Fermenting the submerged dough in a wide container gives the best results. In order to keep the time of testing short an intensive fermentation is required, 6% yeast being used. Sodium chloride is added for dough stability, and 3% of the sugars is added to insure adequate gassing properties. The use of chemical improvers was tried and ammonium phosphate and calcium acid phosphate in combination were found to give good results. It is interesting to note that potassium bromate is not suitable as an improver for this test. Since a newly mixed dough is known to have low expansion properties, the dough is given a period of limited fermentation to develop strength before being expanded under test, and adequate gas production must be assured to give full expansion of the dough. To be of use to the plant breeder the test must require as little flour as possible.

The preliminary testing unit consisted of a 16 oz. wide mouth jar, a large rubber stopper bored to hold a length of graduated precision bore 10 mm. glass tubing graduated in milliliter divisions, and a dough holder attached to the underside of the rubber stopper. A small cruciform deflector prevents the dough from moving up the dough

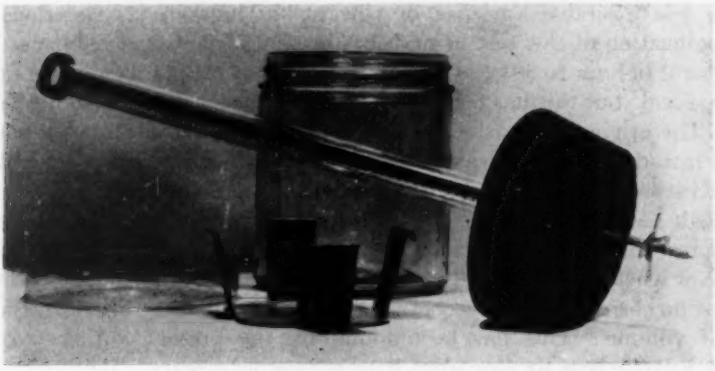

FIG. 1. The apparatus used in making a dough expansion test.

holder after expansion begins. Details are shown in Fig. 1. The underside of the rubber stopper is reamed on a bevel to prevent locking of gas bubbles. A dough carrier of light metal supports the dough inside the jar during the preliminary fermentation. The entire unit is placed in a water bath at 36°C.

The test is conducted in the following manner:

Five grams of flour is placed in a mixing cup and to this is added 2 ml. of salt-sugar solution and 1 ml. of yeast solution, made up as follows:

Salt-sugar solution		*Yeast solution*
Mono ammonium phosphate	0.2 g.	Yeast 12 g.
Calcium acid phosphate	0.3 g.	Water 30 ml.
Sodium chloride	1.0 g.	
Dextrose	3.0 g.	
Sucrose	3.0 g.	
Water	72.1 ml.	

Doughs are mixed for ½ minute to 1¼ minutes according to the strength of the flour. Behavior in mixing is recorded as well as the feel of the dough.

After mixing, the dough is rounded by hand and placed on the carrier in the jar. The jar is partially filled with 0.5% sodium chloride solution, so that a moist atmosphere surrounds the dough when the cover is in place, and the whole is placed in the water bath. Fermentation is allowed to proceed for 45 minutes, after which the dough is rounded up into as compact a ball as possible and impinged on the dough holder against the deflector. The test jar is then filled with 0.5% sodium chloride solution, the stopper firmly fixed in the jar and the solution finally levelled at the zero mark on the graduated tube. The whole unit is then returned to the water bath and expansion allowed to proceed until the dough ball begins to break down. With

the expansion of the dough in the jar, the brine is forced up the graduated tube at a uniform rate until a maximum is reached. At this point large bubbles of gas are first observed rising in the graduated tube and the level of the liquid in the tube begins to fall slightly. There is a distinct misting of the wall of the tube ahead of the rising liquid and when the liquid recedes in the tube a distinct mark is left on the wall at the point of maximum expansion. This mark remains for many minutes after recession begins. This is clearly shown in Fig. 3.

In Fig. 2 the course of expansion is shown for four flours of varying strengths. The rates of expansion are uniform but the time to reach the maximum varies with the strength of the individual flour. The maxima for expansion vary from 8 ml. to 18 ml. and the times from 19 to 38 minutes. In Fig. 3 the appearance of a high strength dough and a low strength dough are shown at peak pressure. A low strength dough frequently disintegrates very quickly at maximum pressure, as shown

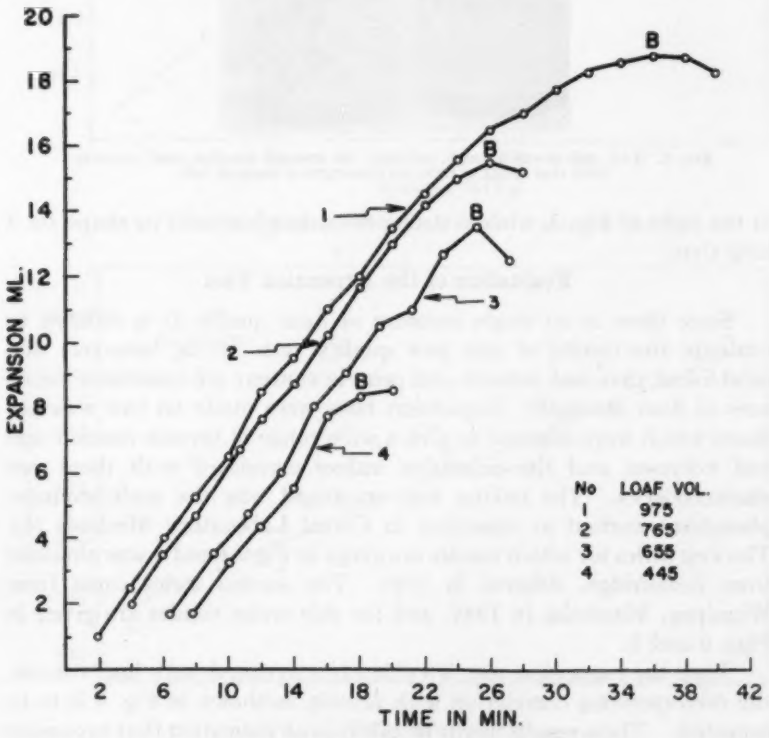

FIG. 2. Graph of expansions in ml. against time in minutes for four doughs showing wide range in quality.

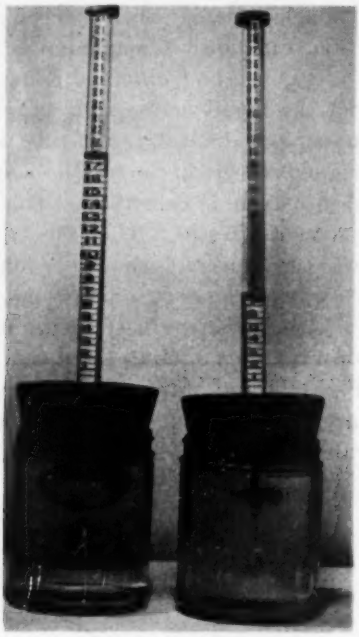

FIG. 3. Left, high strength dough, and right, low strength dough at peak pressures. Note that dough at right has disintegrated plugging tube.

at the right of Fig. 3, while a strong flour dough retains its shape for a long time.

Evaluation of the Expansion Test

Since there is no single measure of flour quality it is difficult to evaluate the results of any new quality test. It is, however, well established that loaf volume and protein content are consistent measures of flour strength. Expansion tests were made on two series of flours which were selected to give a wide range of protein content and loaf volumes, and the expansion values correlated with these two characteristics. The baking test employed was the malt-bromate-phosphate method as described in Cereal Laboratory Methods (6). The first series for which results are given in Figs. 4 and 5 was obtained from Lethbridge, Alberta in 1949. The second series came from Winnipeg, Manitoba in 1949, and for this series results are given in Figs. 6 and 7.

Since the expansion test is evidently correlated with loaf volume, the corresponding correlation with protein as shown in Fig. 4 is to be expected. These results might be taken as an indication that expansion is primarily a measure of protein, especially since r_{13} in Fig. 4, the cor-

relation between loaf volume and protein, is 0.95 and r_{12} in Fig. 5, loaf volume and expansion is 0.88. The results from the Winnipeg series as shown in Figs. 6 and 7, however, show that the expansion figure is related to loaf volume Fig. 7 in spite of a complete absence of correla-

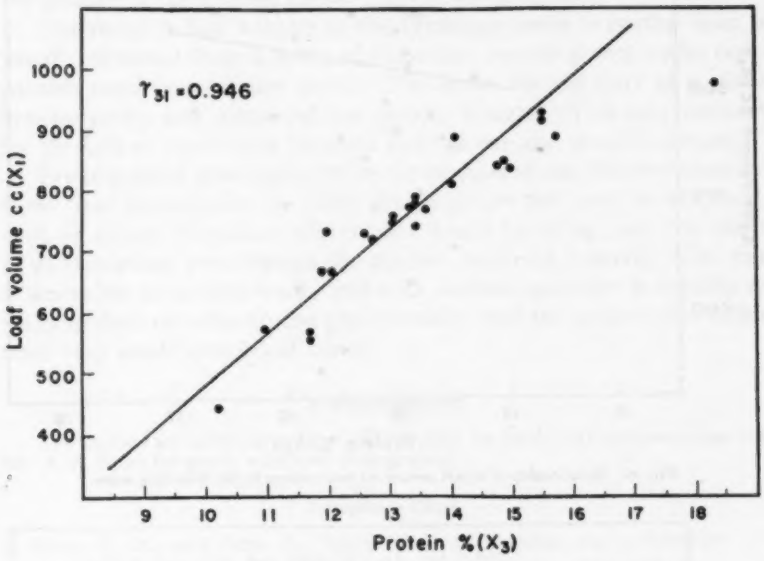

FIG. 4. Relationship of wheat protein to loaf volume in the Lethbridge series.

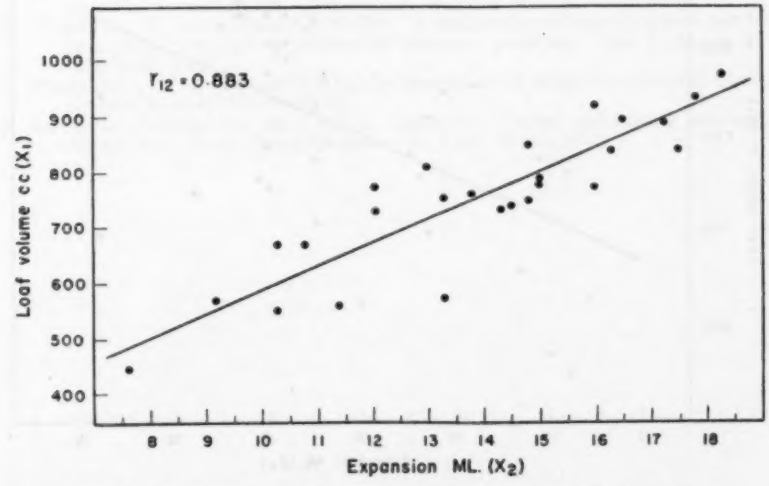

FIG. 5. Relationship of expansion figure to loaf volume in the Lethbridge series.

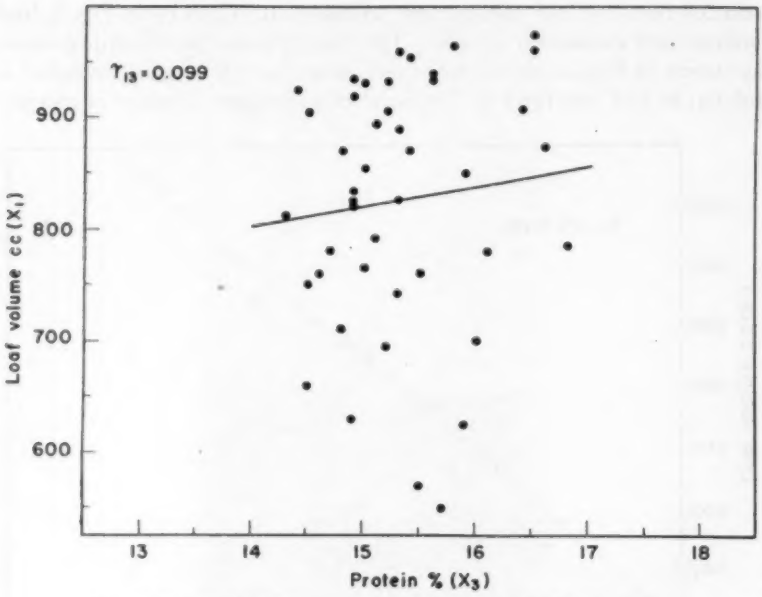

FIG. 6. Relationship of wheat protein to loaf volume in the Winnipeg series.

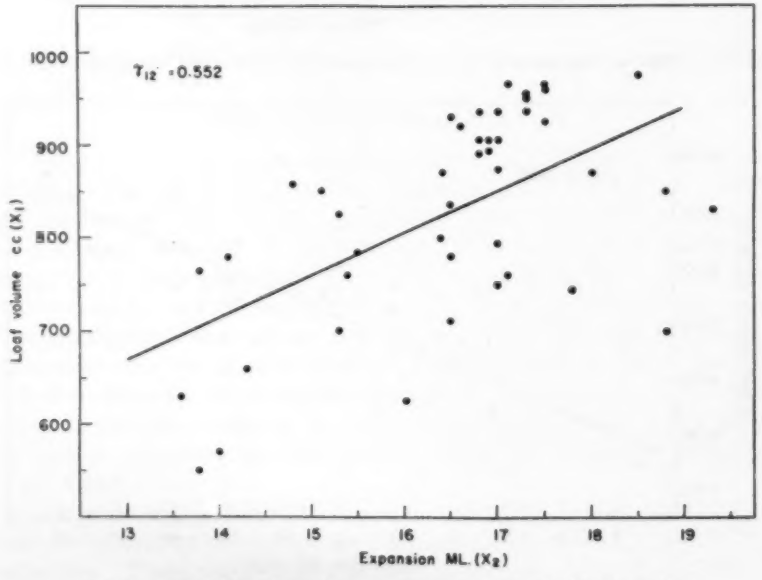

FIG. 7. Relationship of expansion figure to loaf volume in the Winnipeg series.

tion between protein and loaf volume Fig. 6. This group, because it contained high protein wheats of low baking strength, was particularly useful in testing the expansion method against such loaf volumes. The expansion figures followed the loaf volumes, indicating a response to the quality of the protein rather than to its quantity.

The range in loaf volume of the Winnipeg series is greater than is usually obtained from a series of Canadian wheats grown under comparable conditions at one point. The series formed part of a plant breeder group and contained low quality wheats. This may account for the lack of correlation between loaf volume and protein content.

From general observation with the expansion test there are indications that information on other dough properties may be obtained, such as mixing tolerance, absorption, dough handling, and the effect of fermentation, even though the sample employed is small. The test is amenable to routine work, and with careful operation is capable of yielding data of value to the plant breeder and the commercial miller from very small samples of wheat.

Acknowledgment

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INHIBITION OF RESPIRATION AND PRESERVATION OF DAMP WHEAT BY MEANS OF ORGANIC CHEMICALS [1]

SAMUEL A. MATZ and MAX MILNER

ABSTRACT

Deterioration of damp wheat in storage, as measured by increased respiratory rate and free fat-acidity, and by lowered baking quality, can be slightly retarded by certain chemicals. Among those tested, propylene oxide, carbon tetrachloride, and a 1:1 solution of carbon tetrachloride in propylene oxide were the most effective. Propylene oxide seriously reduced the viability of wheat.

None of the treatments counteracted entirely the damaging effects of high-moisture storage even though mold growth was sometimes inhibited. Protracted storage of damp grain under anaerobic conditions appeared to be more damaging to baking quality than storage in an oxygen atmosphere. Chemical treatments might be useful for short-term commercial emergency storage or until the grain can be dried to a safe moisture level.

Previous investigations of the storage properties of damp grain have dealt mostly with the effects of chemical treatment of the grain on mold growth, respiration and heating. Few have dealt with changes in commercial quality due either to mold growth or to chemical treatment. Milner, Christensen, and Geddes (5) investigated 107 compounds for fungistatic action and found that a few of these were effective in inhibiting the respiration of damp grain. Larmour and coworkers (3, 4) investigated carbon tetrachloride as a possible preservative for damp wheat and found that favorable results were obtained when moisture contents were not excessive. Altschul (1) investigated the relative effectiveness of various chemicals as preservatives for cottonseed stored at high moisture contents.

However, Larmour and Bergsteinsson (3) and Swanson (9) have observed that damp wheat treated with a mold inhibitor and stored for an extended time decreases in baking quality, indicating that moisture induces deleterious changes in wheat other than those which can be ascribed to fungi. The present studies were therefore undertaken to determine the effect of certain conditions of storage and the influence of treatment with several chemicals on the respiration, mill-

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ing and baking value, and commercial quality of damp wheat. The studies of Larmour and Bergsteinsson (3) were extended by a quantitative study of the influence of carbon tetrachloride on the respiration and associated deteriorative changes in wheat at several moisture levels when the chemical was applied to the grain in liquid and in vapor form.

Materials and Methods

Over 100 different organic chemicals[2] were given preliminary screening tests to estimate their effectiveness as mold inhibitors. This procedure consisted of mixing 0.1 g. (or 0.1 cc. if liquid) of the chemical with 50 g. of wheat at 20% moisture and storing the mixture at room temperature in a screw-capped glass bottle for 15 days. The time required for the first evidences of mold was noted, and tests for free fat-acidity (6) and germination were made. Several of the most effective mold-inhibiting compounds identified by this procedure were further

[2] Chemicals investigated were:
propylene oxide
alkaterge C
benzyltrimethylammonium chloride
butyl borate
Tris(hydroxymethyl)-nitromethane
2-nitro-2-methyl-1,3-propanediol
hydroxylammonium chloride
2-amino-3-methyl-1,3-propanediol
thiourea
diethyl oxalate
n,n-dicarboxyethylbenezenesulfonamide
kerylbenzene
n,n-dicyanoethylbenzenesulfonamide
n,n-dicyanoethylcyclohexanesulfonamide
ethylene glycol dibenzenesulfonate
kerylbenzyl dichloride
dipropyl ester of n,n-dicarboxyethylbenzenesulfonamide
kerylbenzyl thiocyanate
compound 71
compound 5353
compound 5420
compound 5650
compound 5713
compound 1665
compound 2023
(The preceding seven compounds were experimental preparations of Carbide and Carbon Co. identified only by numbers)
piperonylcyclonene
ethionine
piperonylbutoxide
propylene glycol dipropionate
propylene glycol diacetate
dipropylene glycol diacetate
2,3-dichloro-1,4-naphthoquinone
2-methyl-1,4-naphthaquinone
triethylene glycol diacetate
thioacetic acid
dimethyl dichlorosuccinate
propylene chlorohydrin
parahydroxybenzoic acid
methyl parahydroxybenzoate
ethyl parahydroxybenzoate
propyl parahydroxybenzoate
butyl parahydroxybenzoate
benzil
anisic acid
para-aminobenzoic acid
propylene glycol
ethylene oxide
mesityl oxide
lauric acid
palmitic acid
stearic acid
Arquad S
Arquad 2-C
Neofat 3-R
(The preceding three materials are fatty acid mixtures and/or surface active agents supplied by Armour & Co.)
capric acid
di-tert-butyl peroxide
di-isopropylamine
ethylene chlorohydrin
1,3-dichloropropene 1
dimethyl sulfolane
methallyl alcohol
methallyl chloride
acrolein
allyl alcohol
allyl chloride
epichlorohydrin
hexylene glycol
1,2,3-trichloropropane
beta-dithiocarbamylpropionic acid
Rhodanine
beta-propiolactone
propylene glycol diacetate
dipropylene glycol diacetate
Bioquin-1
anthranilic acid
tripropionin
N-acetyl anthranilic acid
N-acetyl ethylanthranilate
methyl para-aminobenzoate
ethyl para aminobenzoate
N-acetyl methyl para aminobenzoate
N-butyl vanillate
N-propionyl methyl anthranilate
N-benzoyl methyl anthranilate
ethyl anthranilate
propylene dipropionate
ethylene dipropionate
methyl anthranilate
methylene chloride
trichlorethylene
perchlorethylene
chloroform
pentachlorethane
tetrachlorethane
various polymers of propylene oxide

evaluated as respiratory inhibitors and their effect on the commercial quality of wheat and flour was also studied.

Respiration trials were undertaken to compare the efficiency of various wheat treatments as respiration inhibitors. The apparatus used for these studies was similar to that described by Milner and Geddes (7) in which the samples of grain were held at 30°C. and aerated with approximately 2 l. of air per day. Among the chemicals tested for their effectiveness as respiratory inhibitors in wheat were carbon tetrachloride, propylene oxide, ethionine, 2-amino-2-methyl-1,3-propanediol, chlorotrifluoro-ethylmethylether and thiourea, as well as the gaseous flour maturing agents nitrogen trichloride and chlorine dioxide. The solid and liquid chemicals were applied by agitating the dampened wheat with the required amount of chemical in a small air-tight container, while the gaseous compounds were applied by conducting them into an experimental bleacher containing the grain. Constant application of about 2 g. of carbon tetrachloride vapor per day throughout the trial was accomplished by bubbling the incoming air through the liquid chemical.

As a result of the foregoing tests, all but a few of the compounds studied were eliminated from further consideration, either because they were ineffective, or because of their noxious odor, etc. Chemicals found to have desirable characteristics were used in the storage experiment.

For the storage experiment, 3 kg. samples of Pawnee seed wheat from the 1949 harvest, conditioned to 16.7% moisture, were placed in gallon glass jars with screw caps. Five g. (or 5 cc. if liquid) of the appropriate chemical were then added to the wheat in each of four different jars and the mixture was thoroughly agitated before being stored at room temperature. The jar caps were provided with a small hole, ordinarily closed with a rubber stopper, through which a glass tube could be inserted to ventilate the contents with nitrogen or pure oxygen. All chemically treated samples received oxygen. This gas exchange was performed by flushing the grain sample with a strong stream of gas from a cylinder of the compressed gas for a period of about 3 minutes.

At four-week intervals, one jar was removed from each set and the contents subjected to various tests. Baking tests on the flour produced from a part of the sample were conducted in accordance with a straight dough procedure. The following "basic formula" was employed: 100 g. flour (as-is basis), 2 g. yeast, 5 g. sucrose, 2 g. salt, 3 g. shortening and 0.5 g. malted wheat flour. The "bromate formula" consisted of the basic formula plus a quantity of commercial flour improver calculated to give 0.002% potassium bromate. Absorption

percentages were variable and were based on the judgment of the operator performing the mixing.

In view of the effectiveness of propylene oxide in retarding the molding of damp wheat, it seemed advisable to determine the toxicity of this substance to wheat. Previous experiments with such materials had indicated an apparent increase in toxicity as the moisture content increased, and for that reason the influence of moisture content on susceptibility to propylene oxide was investigated.

Tests to determine the toxicity of propylene oxide to wheat were performed by placing a 100 g. sample of wheat in a tin can having a tight lid, adding the required amount of chemical, agitating thoroughly, and storing at room temperature. Samples were withdrawn at convenient times for germination tests by the Seed Laboratory of the Kansas Department of Agriculture.

Results and Discussion

Effect of Carbon Tetrachloride on Respiration. The results of the respirometer trial with wheat containing 18.2% moisture and treated with various concentrations of carbon tetrachloride are given in Table I and Fig. 1 and indicate that, under ordinary conditions, carbon tetrachloride may be an effective depressant of respiration for damp wheat only if comparatively large amounts are used. The delay in the onset of respiratory increases (Fig. 1) is proportional to the amount of carbon tetrachloride added to the wheat initially. This suggests that as long as an effective concentration of carbon tetrachloride is maintained, mold growth is suppressed, but that when this volatile inhibitor is swept out by the air stream passing through the sample,

TABLE I

INHIBITION OF RESPIRATION BY CARBON TETRACHLORIDE IN WHEAT CONTAINING 18.2% MOISTURE

Moisture after trial	Treatment, CCl_4	Respiration mg. CO_2/100 g. dry matter		Germination after trial
		Maximum daily rate attained	Total for trial[1]	
%	%			%
18.2	Original wheat[2]	—	—	79
17.7	None	67.4	436.0	6
17.7	0.03	71.1	424.0	7
17.5	0.1	67.9	359.0	15
17.3	0.2	74.6	298.0	23
17.7	0.3	67.8	245.0	44
17.4	Continuous vapor	16.0	117.0	57

[1] 10-day trial.
[2] Before trial.

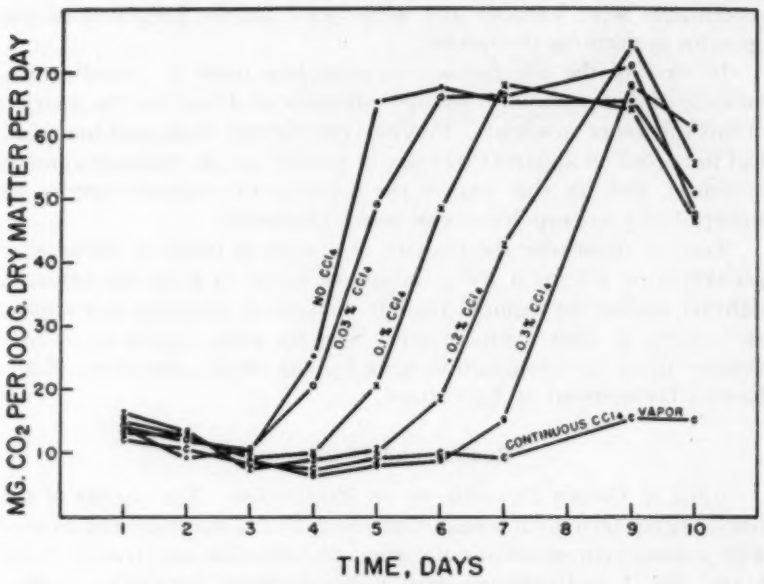

FIG. 1. Influence of various dosages of carbon tetrachloride on the respiratory activity of wheat containing 18.2% moisture.

germination of mold spores and growth of mycelia, which are reflected by increased respiratory activity, are initiated rapidly.

A significant decrease in germination occurred in all samples, even in those which did not respire rapidly. Nevertheless the viability values were roughly proportional to the extent of treatment with the reagent, indicating that suppression of fungal growth was synonymous

TABLE II

INFLUENCE OF CARBON TETRACHLORIDE ON WHEAT STORED AT VARIOUS MOISTURE CONTENTS

Moisture before trial	Moisture after trial	CCl_4 treatment	Respiration mg. CO_2/100 g. dry matter		Germination after treatment
			Maximum daily rate attained	Total for trial[1]	
%	%				%
11	12.8	Continuous vapor	0.10	0.70	93
11	11.3	None	0.00	0.00	97
13	13.0	Continuous vapor	0.40	2.73	92
13	13.1	None	0.23	1.13	94
19	19.2	Continuous vapor	26.1	271.0	95
23	24.0	Continuous vapor	146.0	1,207.0	92

[1] 12-day trial.

with retention of seed viability. The work of Passerini (8) is of interest in this connection. He found that a short immersion of dry wheat in carbon tetrachloride increased germination, but that wheat kept immersed in the chemical for ten months germinated only 4%.

The effect of carbon tetrachloride on the germination and respiration of Comanche variety seed wheat of various moisture contents is shown in Table II. These data show that carbon tetrachloride kept the respiration within reasonable limits and had little deleterious effect on the germination.

The results of the experiment given in Table III show that carbon tetrachloride caused a decrease in respiration and an inhibition of free fat acidity in moist grain. The respiration values were of the same order as those observed in the previous experiment for samples of similar moisture content and treatment.

TABLE III

INFLUENCE OF CARBON TETRACHLORIDE ON RESPIRATION OF WHEAT AT 19%, 21.5% AND 24% MOISTURE CONTENT

Two-stage		CCl_4 treatment	Respiration mg. CO_2/100 g. dry matter		Fat acidity after trial[2]
Moisture before trial	Moisture after trial		Maximum daily rate attained	Total for trial[1]	
%	%				
19.0	18.2	Continuous vapor	21.0	158	18.4
19.0	17.9	None	39.9	217	20.9
21.5	19.7	Continuous vapor	37.4	351	17.5
21.5	19.9	None	69.5	527	26.5
24.0	21.8	Continuous vapor	75.1	716	18.2
24.0	22.0	None	166.0	999	30.6

[1] 10-day trial.
[2] Fat acidity expressed as milligrams potassium hydroxide required to neutralize the fat extracted rom 100 g. of dry material.

These experiments show that carbon tetrachloride is beneficial to wheat that must be stored at moisture levels conducive to mold growth. A slight decrease in germination could be expected, but this effect would, in itself, be of little consequence in wheat destined for milling.

Respiration Studies with Various Mold Inhibitors. The results obtained by treating wheat at about 20% moisture with various respiration inhibitors are presented in Table IV. Propylene oxide was clearly the most effective of the chemicals in inhibiting respiration. Ethionine was slightly more effective than propylene oxide in keeping down the development of free fat acidity. The commonly used maturing agents chlorine dioixde and nitrogen trichloride were only moderately effective as mold inhibitors at relatively high concentrations.

Storage Tests. Results obtained by storing moist grain with various mold inhibitors as shown in Table V show that a constant decrease in moisture content of the samples occurred throughout the test period. Apparently this was due to the periodic flushing of the atmosphere from the jars. The total average decrease in moisture amounted to

TABLE IV

EFFECT OF MOLD INHIBITORS ON THE RESPIRATION OF MOIST WHEAT (100 G. SAMPLES OF WHEAT TEMPERED TO ABOUT 20% MOISTURE)

Treatment	Respiration mg. CO_2/100 g. dry matter		Germination after trial %	Moisture after trial %	Fat acidity after trial[1]
	Maximum daily rate attained	Total for trial			
Trial 1—Six days					
None	134.0	580	44	21.2	60.8
0.5% propylene oxide	15.0	53	0	20.5	41.5
0.5% 2-amino-2-methyl-1,3-propanediol	71.3	358	72	21.0	41.5
0.5% chlorotrifluoroethyl-methyl ether	69.1	380	78	20.9	60.6
0.5% ethionine	75.0	380	64	21.2	38.3
Trial 2—Six days					
None	66.9	341	72	19.3	28.1
0.1% propylene oxide	61.1	196	0	19.1	27.1
0.3% propylene oxide	36.4	73.5	0	19.0	23.7
0.2% thiocarbamide	76.7	259	88	19.3	27.8
0.5% thiocarbamide	55.7	205	85	18.9	26.9
0.2% piperonyl cyclonene	57.0	292	83	19.0	29.0
Trial 3—11 days					
None	73.1	653	27	19.8	40.5
0.037% NCl_3	67.8	562	23	19.7	33.6
0.186% NCl_3	60.2	508	29	19.8	35.7
0.345% ClO_2	70.3	573	25	19.5	38.2
1.725% ClO_2	69.4	585	23	19.7	37.5

[1] Fat acidity expressed as milligrams of potassium hydroxide required to neutralize the fat extracted from 100 g. of dry material.

4.2%. The greatest decrease was in the untreated samples aerated with oxygen and nitrogen and the smallest decrease, as might be expected, occurred in the sealed untreated sample.

A constant and considerable increase in the amount of damaged (sick) kernels occurred over the entire period of the test (Table VI). Aeration of the damp wheat with oxygen considerably decreased the

TABLE V

MOISTURE CONTENT OF DAMP WHEAT AFTER VARIOUS STORAGE INTERVALS

Treatment	Weeks			
	4	8	12	16
	%	%	%	%
Oxygen	16.6	15.6	15.3	11.8
Nitrogen	16.7	15.3	15.5	13.1
Sealed	16.7	15.7	15.4	15.1
0.16% Carbon tetrachloride	16.1	15.1	14.7	14.2
0.16%Propylene oxide in CCl_4	16.2	15.2	15.6	13.4
0.16% Propylene oxide	16.4	15.5	15.1	14.8
0.16% Ethylene chlorohydrin	16.3	15.6	14.9	14.1
0.16% Piperonyl cyclonene	16.3	15.6	15.1	14.2
0.16% Thiourea	16.3	15.0	15.2	14.5

development of damage, while sealing or nitrogen ventilation greatly increased the damage. Of the treated samples, those receiving 5 cc. of propylene oxide in carbon tetrachloride (1:1) had the fewest damaged kernels. All of the dampened samples had decreased in grade from No. 1 Dark Hard Winter to Sample Grade after 16 weeks storage. The grain was musty and objectionable in odor and appearance at that time.

TABLE VI

GRADED COMMERCIAL DAMAGE[1] IN DAMP WHEAT AFTER VARIOUS STORAGE INTERVALS

Treatment	% Damaged kernels			
	4	8	12	16
	Weeks			
Dry control	0.0	0.0	0.0	0.0
Damp wheat, oxygen	1.6	1.1	2.0	3.0
Damp wheat, nitrogen	1.0	1.8	17.0	17.0
Damp wheat, sealed	0.0	1.0	16.0	18.0
Damp wheat, 0.16% carbon tetrachloride	1.3	2.6	4.8	10.0
Damp wheat, 0.16% propylene oxide in CCl_4 (1:1)	1.0	2.6	5.0	6.5
Damp wheat, 0.16% propylene oxide	0.6	1.0	2.0	8.0
Damp wheat, 0.16% ethylene chlorohydrin	1.4	3.4	7.0	15.0
Damp wheat, 0.16% piperonyl cyclonene	0	1.0	8.5	8.5
Damp wheat, 0.16% thiourea	trace	1.2	4.0	6.6

[1] The determination was carried out by the Federal Grain Inspection Office, Kansas City, Missouri.

Free fat acidity increased rapidly in all of the damp samples during the first 12 weeks of storage (Table VII). The rate of increase was considerably lessened during the final four weeks of storage, and in some cases there appeared to be a decrease. Propylene oxide and ethylene chlorohydrin were most effective in retarding the development of free fat acidity, whereas the rate of acidity production in the damp untreated samples was relatively rapid.

TABLE VII

FAT ACIDITY OF DAMP WHEAT AFTER VARIOUS STORAGE INTERVALS

Treatment	Fat acidity[1]			
	4	8	12	16
	Weeks			
Dry control	26.9	26.9	29.2	35.2
Damp wheat, oxygen	45.2	54.9	66.7	60.1
Damp wheat, nitrogen	48.8	54.1	73.4	69.3
Damp wheat, sealed	42.7	47.2	61.0	61.7
Damp wheat, 0.16% carbon tetrachloride	37.3	35.4	48.3	63.7
Damp wheat, 0.16% propylene oxide in CCl_4	31.5	49.0	66.0	57.3
Damp wheat, 0.16% propylene oxide	34.1	42.3	53.4	51.3
Damp wheat, 0.16% ethylene chlorophydrin	28.9	30.8	43.7	40.0
Damp wheat, 0.16% piperonyl cyclonene	38.9	50.9	57.8	58.0
Damp wheat, 0.16% thiourea	41.6	52.7	57.8	43.9

[1] Fat acidity expressed as milligrams potassium hydroxide required to neutralize the fat extracted from 100 g. of dry material.

The mixing time of flours milled from the test wheats tended to increase with the duration of storage as indicated in Table VIII. Such an increase was not observed in farinograms for the flour milled from the control sample stored in the dry condition. Also, as the length of storage increased, the mixing curves of these doughs showed little breakdown and the farinograph curves flattened out. This change did not occur in flour from wheat treated with thiourea.

When these flours were baked (Table IX) there appeared a most striking drop in bromate response. With some, negative response to bromate was obtained, but usually a slight decrease in specific volume of the basic formula loaves resulted from storage of the wheat in damp condition. Some of the chemical treatments appeared to cause an increase in the specific volume of the basic formula loaves. Most effective in this respect were piperonyl cyclonene and propylene oxide

TABLE VIII

FARINOGRAPH MIXING TIME OF FLOURS MILLED FROM DAMP WHEAT AFTER VARIOUS STORAGE INTERVALS

Treatment	Farinograph mixing time (minutes)			
	4	8	12	16
	Weeks			
Dry control	4.0	4.5	4.0	—
Damp wheat, oxygen	4.5	5.0	6.5	5.5
Damp wheat, nitrogen	4.5	6.5	9.0	7.5
Damp wheat, sealed	5.0	5.5	6.5	7.0
Damp wheat, 0.16% carbon tetrachloride	5.0	6.0	6.0	7.5
Damp wheat, 0.16% propylene oxide in CCl_4	6.5	7.5	8.0	8.0
Damp wheat, 0.16% propylene oxide	5.0	7.0	9.0	8.0
Damp wheat, 0.16% ethylene chlorohydrin	5.0	7.0	8.0	6.0
Damp wheat, 0.16% piperonyl cyclonene	4.5	6.5	6.0	7.5
Damp wheat, 0.16% thiourea	4.0	6.0	6.5	6.0

TABLE IX

BAKING CHARACTERISTICS OF DAMP WHEAT AFTER 16 WEEKS OF STORAGE AS INFLUENCED BY VARIOUS TREATMENTS

Treatment	Dough formula	Specific volume	Grain score
Dry control	basic	5.37	85
	bromate	7.62	90
Damp wheat, oxygen	basic	5.42	87
	bromate	6.62	80
Damp wheat, nitrogen	basic	5.84	82
	bromate	5.19	85
Damp wheat, sealed	basic	4.79	85
	bromate	6.16	85
Damp wheat, 0.16% carbon tetrachloride	basic	5.45	87
	bromate	6.73	87
Damp wheat, 0.16% propylene oxide in CCl_4	basic	6.01	80
	bromate	6.20	82
Damp wheat, 0.16% propylene oxide	basic	4.98	85
	bromate	5.67	80
Damp wheat, 0.16% ethylene chlorohydrin	basic	5.64	85
	bromate	5.87	87
Damp wheat, 0.16% piperonyl cyclonene	basic	6.55	85
	bromate	6.58	90
Damp wheat, 0.16% thiourea	basic	5.43	80
	bromate	5.36	85

in carbon tetrachloride. Flours from all of the wheats stored 16 weeks in damp condition gave loaves that were grayish in color and musty in odor.

Effect of Propylene Oxide on the Viability of Wheat. The data in Table X indicate that addition of 0.2% or more of propylene oxide to wheat causes an immediate drop of the germination to very low values. This phenomenon is apparent even in wheat at low moisture contents but an increase in moisture increases the effect.

Results obtained in this investigation indicate that it is possible to retard the storage deterioration of damp wheat by application of certain fungistatic chemicals. However, none of the compounds employed was able to overcome all the damaging effects of excess moisture which were greatest in untreated samples stored under anaerobic conditions. The results suggest that chemicals might be satisfactory for use on a commercial scale as a short-time emergency measure, as an adjunct to drying, to prevent the occurrence of heat damage in wheat received at the storage point with an unsafe moisture content. Possible deleterious side effects of the preservative should be carefully considered before it is applied on a large scale. The deleterious changes which occur in damp grain in spite of the presence of fungistatic agents, and particularly in the absence of oxygen, needs further clarification before chemical preservation of crops can be recommended as a practical measure.

TABLE X

EFFECT OF PROPYLENE OXIDE ON THE VIABILITY OF WHEAT
(ORIGINAL GERMINATION 96%)

Treatment	Per cent germination				
	Days of storage				
	2	4	6	9	11
11.1% moisture, 2% propylene oxide	2	0	0	2	0
11.1% moisture, 0.2% propylene oxide	10	8	12	5	7
11.1% moisture, no further treatment	96	—	—	—	97
15% moisture, no further treatment	97	87	94	92	93
15% moisture, 0.2% propylene oxide	0	0	0	0	0
20% moisture, no further treatment	96	92	88	81	53
20% moisture, 0.2% propylene oxide	0	0	0	0	0

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INFLUENCE OF WHEAT VARIETY, MALT, AND SHORTENING ON THE CHARACTERISTICS OF CHEMICALLY LEAVENED BISCUITS [1,2]

HOMER R. ELLING AND MAX MILNER

ABSTRACT

Flours from hard winter wheat varieties generally considered satisfactory for bread production yielded biscuits of good quality, with the exception of the variety Red Chief. In this study, score values for crumb compressibility, volume, and specific lightness were employed as well as other quality factors. All soft wheat varieties tested except Purkof gave satisfactory results. Supplementation of hard and soft wheat flours with malted wheat flour or extracts thereof resulted in some improvement in biscuit characteristics up to an optimum level of supplementation, which was followed by a decrease in quality at higher levels. The improvement noted appeared to be associated with the alpha-amylase component. Optimum shortening content was found to be significantly correlated with protein content and viscosity only for hard wheat flours or when values for hard and soft types are combined. Crumb compressibility and crumbliness showed no relationship in the hard and soft wheat flours tested nor did protein content appear to have any influence on these values.

The major objective attained by various committees of the American Association of Cereal Chemists which have dealt with the testing of self-rising flours, has been the development of a reliable biscuit baking test (1). Other valuable information obtained in these studies includes clarification of the influence of absorption and pH (5), flour

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[2] Contribution No. 182, Department of Milling Industry, Kansas Agricultural Experiment Station, Manhattan, Kansas.
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granulation (4), shortening requirement (7), the relation of absorption to shortening requirement (6), and the influence of leavening agents on gluten properties (8).

The present studies were undertaken to clarify further the relationship of certain flour characteristics and treatments to biscuit quality. Factors studied included the influence of wheat variety on flour quality for biscuit purposes, the effect of the addition of malted wheat flour as well as extracts thereof, the relation of flour viscosity and protein content to shortening requirement, and the influence of some of these variables on the crumbliness and compressibility of the biscuit crumb.

Materials and Methods

Hard and soft wheat flours obtained from commercial sources as well as a number of special Buhler-milled flours were employed. For the study of the influence of wheat varieties on biscuit quality, a series of hard red wheat variety composites, obtained from the Federal Hard Wheat Quality Laboratory at Manhattan, Kansas were experimentally milled. A similar series of flour was milled from soft wheat varieties obtained from the Federal Soft Wheat Laboratory at Wooster, Ohio.

For the study of the influence of malt on baking quality, hard and soft wheat flours were supplemented to various malt levels with a commercial malted wheat flour. In a similar series, supplementation was accomplished with an aqueous extract of the malt flour, prepared by treating 20 g. of malted wheat flour with 200 ml. of distilled water and shaking periodically for one hour before filtering. To determine whether the effect of the malt supplement lay in the amylase or the protease fraction, one series was supplemented with various concentrations of malt extract in which the proteolytic activity has been removed by the procedure described by Dirks and Miller (3).

For the study of the relationship of protein content to shortening requirement, 27 flours of widely varying characteristics were employed. In addition to the commercially milled hard and soft wheat flours, a number of hard wheat flours were milled from wheat composites of different protein levels. In this group also was a series of hard and soft wheat flours supplemented with undenatured gum gluten[3]; another group consisted of the commercial flours diluted to lower protein levels with wheat starch. A number of the flour samples were obtained from pure variety hard and soft wheats.

All analytical values were obtained by procedures outlined in Cereal Laboratory Methods (1) unless otherwise specified. The baking method employed for biscuits was essentially that given in that volume. A minor variation involved rolling the dough to a thickness

[3] Supplied by the Northern Regional Research Laboratory, Peoria, Illinois.

of ¼ in. instead of ⅜ in. This procedure was found to yield biscuits of greater uniformity. A more comprehensive scoring method than that of the prescribed procedure also was adopted. The score card used was as follows:

Score factor	*Maximum numerical score*
External appearance	12
Texture	13
Compressibility	15
Flavor	15
Crust color	10
Crumb color	15
Lightness	10
Volume	10

Compressibility score was obtained with a Bloom Gelometer as described later and the experimental values were transferred to the numerical scale of 1 to 15 by appropriate statistical methods, on the assumption that the experimental measurements were normally distributed (9). By similar treatment the biscuit volume and lightness (specific volume) data were transferred to the score card scale indicated.

Values for specific volume and specific lightness are derived as follows:

$$\text{Specific volume} = \frac{\text{volume of 7 biscuits}}{\text{dough wt. of 7 biscuits}} \times 20$$

$$\text{Specific lightness} = \frac{\text{volume of 7 biscuits}}{\text{baked wt. of 7 biscuits}} \times 20$$

The Amylograph viscosity tests on flour-water suspensions were carried out by the procedure outlined by Anker and Geddes (2).

Crumbliness was determined with the specially designed instrument shown in Fig. 1. This instrument consisted of a reciprocating arm driven from a series of belted pulleys by an electric motor. To the end of the arm was clamped a hollow cylindrical container assembled from two threaded sections, with a ¼-in. mesh screen inserted between the sections. The speed of the reciprocating arm as well as the length of throw could be varied. To carry out a crumbliness determination a weighed quantity of crumb prepared in a mitre box to definite dimensions was placed in the container above the screen, and after the container halves were screwed together the container was clamped into position on the reciprocating arm. After the required time of shaking, the amount of crumb passing the screen was weighed to provide the crumbliness index. Generally this test was conducted by shaking for 8 minutes with the crumb obtained one hour after removal of the biscuits from the baking oven.

Crumb compressibility was determined using the Bloom Gelometer, the values obtained being the weight in grams required to produce a penetration of 4 mm. in a slice 0.5 in. thick.

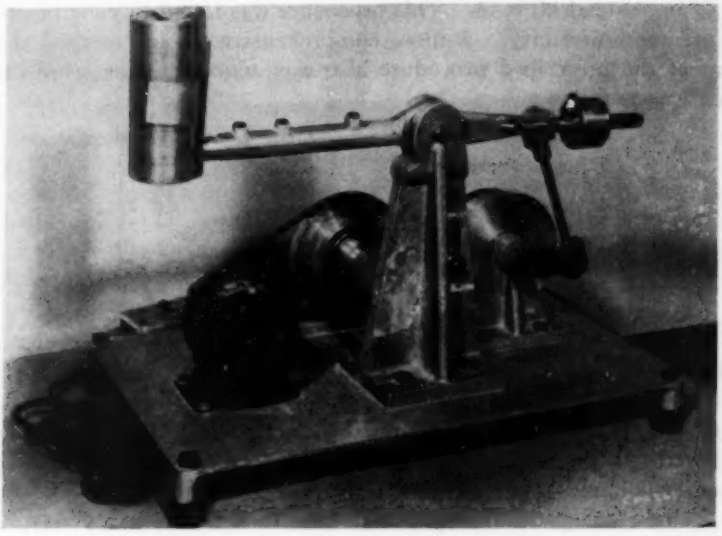

TABLE I

RELATION OF WHEAT VARIETY TO BISCUIT PROPERTIES.
I. HARD WINTER WHEAT VARIETIES

	Variety				
	Hard Standard[1]	Wichita[2]	Early Blackhull[2]	Red Chief[2]	Pawnee[2]
Quality factors					
Optimum absorption, %	65.0	67.5	62.5	77.5	70.0
Optimum shortening, %	15.0	12.5	12.5	17.5	15.0
Specific volume	44.5	48.1	59.7	41.2	47.6
Specific lightness	58.0	61.8	64.5	54.3	60.1
Gelometer compressibility	127.7	111.0	128.5	152.6	132.0
pH	7.4	7.4	7.4	7.37	7.45
Score values					
External appearance	12	11	11	9	12
Texture	13	13	13	12	13
Compressibility	7	12	7	2	6
Flavor	15	15	15	15	15
Crust color	10	10	10	10	10
Crumb color	15	15	15	15	15
Lightness	4	8	9	2	7
Volume	4	8	9	1	7
Total score	80	92	89	66	85

[1] Mean values derived from 11 replicate determinations.
[2] Mean values derived from 4 replicate determinations.

Results and Discussion

Influence of wheat variety on biscuit quality. The biscuit making quality of flours experimentally milled from a number of wheat varieties is summarized in Tables I and II for hard and soft wheat types respectively. These data show that certain varieties are superior to others for biscuit making. Thus among the hard wheat varieties, acceptable biscuits are produced by Wichita, Early Blackhull and Pawnee, which are closely grouped in the order of quality when ranked by total quality score. The variety Red Chief, however, yielded

TABLE II

RELATION OF WHEAT VARIETY TO BISCUIT PROPERTIES.
II. SOFT WHEAT VARIETIES

	Variety						
	Soft Standard[1]	Black-hawk[2]	Trum-bull[2]	Purkof[3]	Clarkan[2]	Thorne[3]	American Banner[2]
Quality factors							
Optimum absorption, %	60.0	62.5	62.5	67.5	65.0	62.5	65.0
Optimum shortening, %	10.0	10.0	10.0	12.5	10.0	12.5	10.0
Specific volume	51.3	49.7	48.1	48.3	49.2	47.6	45.6
Specific lightness	64.1	61.2	62.4	59.8	63.2	58.9	60.2
Gelometer compressibility	81.7	83.0	80.0	103.0	80.0	74.0	79.0
pH	7.3	7.4	7.4	7.3	7.37	7.4	7.3
Score values							
External appearance	12	11	11	11	11	12	12
Texture	13	13	12	11	11	12	13
Compressibility	9	8	9	2	9	12	10
Flavor	15	15	15	15	15	15	15
Crust color	10	10	10	10	10	10	10
Crumb color	15	15	15	15	15	15	15
Lightness	6	3	5	2	5	2	3
Volume	7	5	3	3	4	2	1
Total score	87	80	80	69	80	78	79

[1] Mean values derived from 34 replicate determinations.
[2] Mean values derived from 5 replicate determinations.
[3] Mean values derived from 4 replicate determinations.

biscuits of definitely inferior characteristics. This flour had an absorption approximately 11% higher than the average of that for the other flours tested, but most of the other quality criteria were inferior. The low rank of this variety in biscuit making quality is similar to that which it holds for breadmaking purposes.

With the marked exception of Purkof, the other soft wheat varieties tested, namely Blackhawk, Trumbull, Clarkan, Thorne, and American Banner, yielded acceptable biscuits with similar score values. The average score for the acceptable biscuits produced from the hard wheat

varieties was approximately 6 score points better than that from the average for the satisfactory soft wheat varieties, with the standards included in both cases.

Effect of wheat malt on biscuit properties. The influence of malted wheat flour on the biscuit making quality of the standard hard and soft wheat flours is shown in Tables III and IV together with their maltose values and maximum Amylograph viscosities.

The results with hard winter flour show that biscuit quality is improved with the addition of malted wheat flour although this improvement is not always regular, as indicated by total score. External appearance was not changed whereas crumb texture showed improvement with 0.5, 1.0, and 1.5% malt; at higher increments the texture became progressively coarser. A definite trend in crumb compressibility was not secured. The crust color of the biscuits became progressively darker with the addition of increasing quantities of malted wheat flour.

Biscuit volume improved with increments of malt flour up to 2.5%, but additions beyond this point caused the volume to decrease. Light-

TABLE III

INFLUENCE OF ADDED MALTED WHEAT FLOUR ON BISCUIT PROPERTIES.
I. COMMERCIAL HARD WHEAT FLOUR

	Per Cent Malted Wheat Flour						
	0.0[1]	0.5[2]	1.0[2]	1.5[2]	2.5[2]	5.0[2]	10.0[2]
Flour characteristics							
Maltose value	244	328	367	406	445	455	555
Max. amylograph vis.	280	140	130	110	90	70	50
Quality factors							
Optimum absorption, %	65.0	65.0	65.0	65.0	65.0	65.0	65.0
Optimum shortening, %	15.0	15.0	15.0	15.0	15.0	15.0	15.0
Specific volume	44.5	44.9	46.8	48.6	48.0	45.6	45.9
Specific lightness	60.0	59.5	59.2	59.7	61.2	61.8	59.5
Gelometer compress.	127.5	123.7	110.0	122.0	145.0	131.0	108.0
pH	7.25	7.3	7.3	7.3	7.35	7.35	7.35
Score values							
External appearance	12	12	12	12	12	12	12
Texture	12	13	13	13	12	12	11
Compressibility	7	9	12	9	3	6	13
Flavor	15	15	15	15	15	15	15
Crust color	5	6	7	8	9	10	10
Crumb color	15	15	15	15	15	15	15
Lightness	7	6	4	7	7	8	6
Volume	4	4	6	8	8	5	5
Total score	82	80	84	87	81	83	87

[1] Mean values derived from 11 replicate determinations.
[2] Mean values derived from 6 replicate determinations.

TABLE IV

INFLUENCE OF ADDED MALTED WHEAT FLOUR ON BISCUIT PROPERTIES.
II. COMMERCIAL SOFT WHEAT FLOUR

	Per Cent Malted Wheat Flour						
	0.0[1]	0.5[2]	1.0[2]	1.5[2]	2.5[2]	5.0[2]	10.0[2]
Flour characteristics							
Maltose value	96	151	195	231	265	315	415
Max. amylograph visc.	740	150	100	80	80	40	30
Quality factors							
Optimum absorption, %	60.0	60.0	60.0	60.0	60.0	60.0	60.0
Optimum shortening, %	10.0	10.0	10.0	10.0	10.0	10.0	10.0
Specific volume	51.3	52.7	54.5	52.5	51.8	50.3	50.0
Specific lightness	64.1	65.3	66.3	64.1	66.7	63.4	63.3
Gelometer compress.	81.7	81.0	60.0	80.0	67.8	47.0	73.7
pH	7.3	7.25	7.25	7.25	7.3	7.3	7.3
Score values							
External appearance	12	12	12	12	12	12	12
Texture	13	13	13	13	12	11	11
Compressibility	9	9	15	9	13	15	12
Flavor	15	15	15	15	15	15	15
Crust color	8	8	8	9	10	10	10
Crumb color	15	15	15	15	15	15	15
Lightness	6	8	8	6	9	6	6
Volume	7	8	9	8	7	5	5
Total score	85	88	95	87	93	89	86

[1] Mean values derived from 34 replicate determinations.
[2] Mean values derived from 5 replicate determinations.

ness score was variable throughout this series and a slight increase in pH was noted with successive additions of malt.

The result obtained with the soft wheat flour was similar to that with the hard wheat type. The total score was highest with 1% malted wheat flour but fell off with higher increments. Texture became significantly coarser at values above 1.5% and crust color became progressively darker as the malted wheat flour was increased. Volume score increased with additions up to 1% but decreased with higher increments.

The influence of malt was further investigated by adding aqueous extracts of the malted wheat flour to the standard soft wheat flour. The results of this study are shown in Table V and indicate a similar trend to that shown when malted wheat flour alone was used. External appearance of all the biscuits of this series was satisfactory but crumb texture became progressively more open with the addition of the extract. The total score suggested that the best biscuits were obtained by supplementation with 20 ml. of malted wheat flour extract.

Inactivation of the proteolytic enzymes of the malted wheat flour extract yielded the results shown in Table VI, which are very similar to those obtained with the untreated extract. The maximum improvement was obtained with the first 20 ml. addition. In this case, also, the texture suffered slightly with addition of the extract while compressibility increased. Further addition, however, caused a marked reduction in the crumb compressibility.

TABLE V

INFLUENCE OF MALTED WHEAT FLOUR EXTRACT ON BISCUIT PROPERTIES (COMMERCIAL SOFT WHEAT FLOUR)

	ml. Malted Wheat Flour Extract				
	0[1]	20[2]	30[2]	40[2]	100[2]
Flour characteristics					
Maltose value	96	195	210	225	500
Maximum amylograph viscosity	740	200	180	150	120
Quality factors					
Optimum absorption, %	60.0	60.0	60.0	60.0	60.0
Optimum shortening, %	10.0	10.0	10.0	10.0	10.0
Specific volume	51.3	52.0	49.6	48.5	48.8
Specific lightness	64.1	64.9	62.8	59.3	60.6
Gelometer compressibility	81.7	62.0	66.0	93.0	92.0
pH	7.35	7.4	7.45	7.3	7.25
Score values					
External appearance	12	12	12	12	12
Texture	13	12	11	11	11
Compressibility	9	14	14	4	5
Flavor	15	15	15	15	15
Crust color	9	9	9	10	10
Crumb color	15	15	15	15	15
Lightness	6	7	5	2	3
Volume	7	7	4	3	4
Total score	86	91	85	72	77

[1] Mean values derived from 34 replicate determinations.
[2] Mean values derived from 4 replicate determinations.
[3] Mean values derived from 3 replicate determinations.

In general, the improvement in volume of biscuits due to the use of malt was accompanied by a less pronounced improvement of the interior of the biscuit, and approximately 1% malt flour yielded optimum results. The fact that compressibility scores seem to improve with the addition of malt up to an optimum suggests that a certain amount of starch modification due to the amylase activity may be beneficial. The results indicate that amylase enzymes are active in the short period between mixing of a biscuit dough and attainment of temperature of inactivation in the oven.

Relation of shortening requirement to viscosity and protein content. The optimum shortening requirements, as determined by the standard baking procedure for 16 hard and 11 soft wheat flours of widely varying characteristics, are indicated in Table VII together with the viscosity and protein values for these flours.

The correlations of optimum shortening content with flour viscosity and protein content were statistically highly significant (beyond the 1% level) only in the case of the hard wheat flours. The corresponding

TABLE VI

INFLUENCE OF PROTEINASE-INACTIVATED MALTED WHEAT FLOUR EXTRACT ON BISCUIT PROPERTIES (COMMERCIAL SOFT WHEAT FLOUR)

	ml. Proteinase-Inactivated Malted Wheat Flour Extract				
	0[1]	20[2]	30[2]	40[2]	100[2]
Flour characteristics					
Maltose value	96	153	168	178	255
Maximum amylograph viscosity	740	210	190	180	140
Quality factors					
Optimum absorption, %	60.0	60.0	60.0	60.0	60.0
Optimum shortening, %	10.0	10.0	10.0	10.0	10.0
Specific volume	51.3	54.6	52.0	51.3	50.1
Specific lightness	64.1	63.9	66.0	66.8	63.5
Gelometer compressibility	81.7	66.0	98.0	97.0	97.0
pH	7.3	7.37	7.37	7.3	7.2
Score values					
External appearance	12	12	12	12	12
Texture	13	12	11	11	11
Compressibility	9	14	3	3	3
Flavor	15	15	15	15	15
Crust color	9	9	9	10	10
Crumb color	15	15	15	15	15
Lightness	6	6	8	9	6
Volume	7	9	7	7	5
Total score	86	92	80	82	77

[1] Mean values derived from 34 replicate determinations.
[2] Mean values derived from 3 replicate determinations.

correlation coefficients for soft wheat flours were not statistically significant at the 5% level. However when the values for the hard and soft were combined the correlations for both sets of values were highly significant.

Crumbliness and Compressibility. Ten replicate determinations of crumbliness were carried out with hard and soft wheat biscuit crumbs at three shaking times (1, 5, and 8 minutes) and six cooling times (0.5, 1.0, 3.0, 6.0, 24, and 48 hours). Based on these tests a standard procedure of one hour cooling and 8 minutes of shaking was adopted

to furnish the crumbliness index. The crumbliness index and the crumb compressibility obtained with the Bloom Gelometer were determined for biscuits baked with shortening contents and absorption values which were shown to yield optimum quality characteristics. Biscuits representing the 27 different flours used in this study were investigated. The data for crumbliness, compressibility, and protein content are shown in Table VIII.

TABLE VII

RELATION OF OPTIMUM SHORTENING REQUIREMENT TO VISCOSITY AND PROTEIN CONTENT

Flour Protein[1]	Viscosity[2]	Shortening
%	°MacMichael	%
	Hard wheat flours	
9.2	137	15.0
7.8	67	10.0
9.3	101	15.0
10.6	140	15.0
12.2	183	17.5
14.0	234	20.0
7.2	49	10.0
8.4	87	12.5
11.4	169	17.5
13.0	175	17.5
10.5	137	12.5
10.0	142	12.5
10.3	137	17.5
11.6	170	15.0
9.3	103	15.0
9.5	96	15.0
	Soft wheat flours	
7.4	60	10.0
7.0	45	10.0
8.0	77	12.5
9.3	95	12.5
11.0	114	15.0
8.4	53	10.0
10.2	84	10.0
9.7	126	10.0
8.0	84	12.5
8.3	77	10.0
9.0	133	12.5

Correlation Coefficients

Visc. (20 g.) × shortening % (hard wheat flour)	= 0.83**
Visc. (20 g.) × shortening % (soft wheat flour)	= 0.50
Visc. (20 g.) × shortening % (all flours)	= 0.83**
Protein % × shortening % (hard wheat flour)	= 0.85**
Protein % × shortening % (soft wheat flour)	= 0.45
Protein % × shortening % (all flours)	= 0.81**

[1] 14% moisture basis.
[2] 20 g. flour, 14% moisture basis.
** Double asterisks indicate that these sample correlations are statistically significant beyond the 1% level. The other correlations given are non-significant at the 5% level.

TABLE VIII

RELATION OF CRUMBLINESS AND COMPRESSIBILITY TO FLOUR PROTEIN

Protein[1]	Crumbliness Index	Compressibility
		g.
	Hard wheat flours	
9.2	48.9	127.7
7.8	67.4	113.0
9.3	71.3	119.0
10.6	66.3	161.0
12.2	67.3	166.0
14.0	62.2	183.0
7.2	65.8	168.4
8.4	65.7	152.0
11.4	63.2	150.0
13.0	71.2	139.0
10.0	40.8	111.0
10.5	44.0	128.5
10.3	41.7	152.6
11.6	31.9	132.0
9.3	41.9	98.0
9.5	44.3	81.7
	Soft wheat flours	
7.4	45.3	81.7
7.0	50.6	86.0
8.0	55.5	81.0
9.3	63.3	94.0
11.0	47.7	72.0
10.2	34.3	83.0
9.7	35.6	80.0
9.0	54.3	103.0
8.3	44.4	80.0
8.0	53.0	74.0
8.4	57.0	79.0

Correlation Coefficients

Compressibility × crumbliness (hard wheat flour)	= + 0.46
Compressibility × crumbliness (soft wheat flour)	= + 0.33
Compressibility × crumbliness (all flours)	= + 0.48*
Protein content × crumbliness (soft wheat flour)	= − 0.32
Protein content × crumbliness (hard wheat flour)	= + 0.02
Protein content × crumbliness (all flours)	= + 0.07

[1] 14% moisture basis.
* Asterisk indicates that this correlation is statistically significant beyond the 5% level. The other correlations are non-significant at the 5% level.

No statistically significant correlation between compressibility and crumbliness was found at the 5% level for either hard or soft wheat flours, although the value for hard wheat flour does border on significance. However when the data for hard and soft types are combined, the correlation obtained is significant at the 5% level.

Similar correlations for protein content and crumbliness yielded values of no significance. It would appear from this study that factors other than protein content are primarily responsible for biscuit crumbliness.

Acknowledgment

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CHEMICAL DETERMINATION OF CALCIUM PANTOTHENATE [1,2]

C. R. SZALKOWSKI, W. J. MADER AND H. A. FREDIANI

ABSTRACT

A chemical method for the determination of calcium pantothenate has been developed which requires a sample weight containing about 0.2 g. of the vitamin and which is suitable for the analysis of many vitamin preparations and feed enrichment mixtures.

Hydrolytic cleavage of pantothenic acid in alkaline or acid media results in the formation of beta alanine and alpha, alpha-dihydroxy-beta, beta-dimethylbutyric acid. Beta alanine when treated with potassium permanganate in the presence of potassium bromide under properly regulated conditions yields an insoluble compound upon treatment with 2,4-dinitrophenylhydrazine. The ratio between the amount of this product and the hydrolyzed pantothenate oxidized is constant and can be estimated by dissolving the dinitrophenylhydrazone in pyridine, diluting with sodium hydroxide and measuring the resulting blue color spectrophotometrically at 570 millimicrons. Beer's law is followed over a suitable concentration range.

Interference from niacin, niacinamide, thiamine, vitamin B_6, and soya flour is removed by chromatographing the aqueous solution of the sample on aluminum oxide columns. Ascorbic acid, however, interferes and has not been successfully removed chromatographically as yet.

Calcium pantothenate is one of the ingredients currently being added to various vitamin preparations and feed enrichment mixtures.

[1] Received November 9, 1950.
[2] A contribution from the Chemical Control Division, Merck & Co., Inc., Rahway, N. J.

Up to the present, control of this constituent in such mixtures has been based on microbiological assays (1, 2, 3) which require from 24 to 72 hours for completion. Recently Schmall and Wollish (4) have described a colorimetric method based on hydrolysis of the pantothenate to pantoyl lactone, formation of hydroxamic acid by addition of hydroxylamine hydrochloride in alkaline solution and the development of a purple color with ferric chloride. These latter authors include an adequate bibliography on the analysis of panthenol and pantothenates.

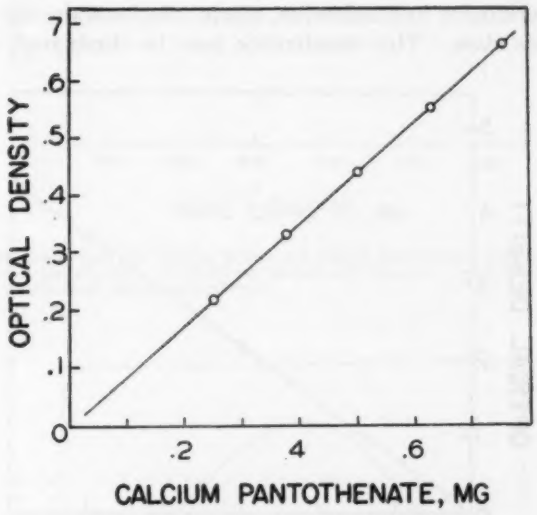

FIG. 1. Calibration curve using alkaline hydrolysis.

Hydrolytic cleavage of pantothenic acid in alkaline or acid media results in the formation of beta alanine and alpha, alpha-dihydroxy-beta, beta-dimethyl butyric acid. Beta alanine when oxidized with potassium permanganate in the presence of potassium bromide under properly regulated conditions yields an insoluble hydrazone with 2,4-dinitrophenylhydrazine. The ratio between the amount of hydrazone formed and the hydrolyzed pantothenate oxidized is constant. It may be estimated by dissolving the dinitrophenylhydrazone in pyridine, diluting with sodium hydroxide and measuring the resulting blue color spectrophotometrically at 570 mu. It may be seen from Figs. 1 and 2 that Beer's law is followed over a suitable concentration range, with either acid or alkaline hydrolysis. Thus, for pure calcium pantothenate, as well as in some mixtures, either the acid or alkaline hydrolysis may be used with an appropriate calibration curve. How-

ever, in certain mixtures (for example, with soya flour) acid hydrolysis is essential.

The specificity of this reaction with respect to the other vitamins and organic acids usually encountered has been studied. The presence of relatively large amounts of such compounds as acetic, lactic, tartaric, glycollic and succinic acids, alpha alanine, ethyl alcohol and riboflavin has been found not to affect the pantothenate estimation. Compounds which yield insoluble dinitrophenylhydrazones after oxidation with potassium permanganate, however, do interfere. Compounds such as carbohydrates, ascorbic acid, thiamine hydrochloride, pyridoxine hydrochloride, niacin, niacinamide and soya flour fall into this class. This interference may be eliminated, except for

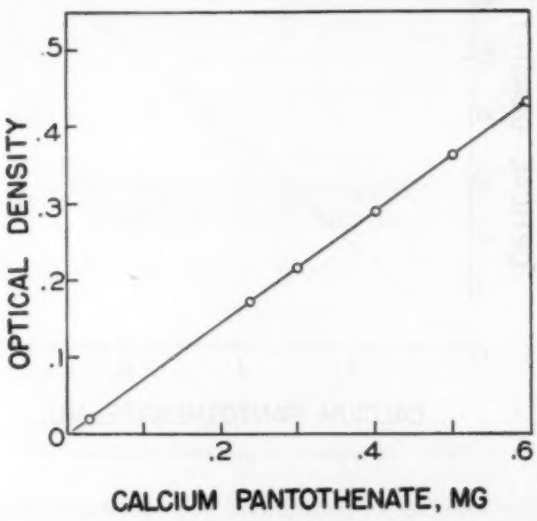

FIG. 2. Calibration curve using acid hydrolysis.

ascorbic acid, by chromatographic isolation of the calcium pantothenate. Figure 3 shows the absorption curve obtained for alkaline hydrolyzed calcium pantothenate, beta alanine and unhydrolyzed calcium pantothenate. This curve suggests a possibility of determining beta alanine in pantothenate without separation.

Figure 4 indicates the absorbancies produced by various weights of thiamine hydrochloride, ascorbic acid, and niacin after alkaline hydrolysis. These materials were not chromatographed.

In Fig. 5 are shown the absorbancies of acid hydrolyzed material. To measure calcium pantothenate precisely, it is obviously necessary that it be separated from constituents such as these.

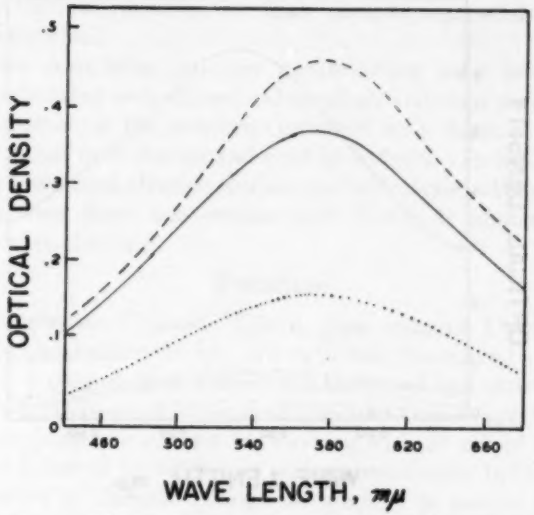

FIG. 3. Absorption curve for alkaline hydrolyzed calcium pantothenate, beta alanine, and unhydrolyzed calcium pantothenate. ——0.375 mg. calcium pantothenate. ——0.188 mg. beta alanine. ....0.375 mg. unhydrolyzed calcium pantothenate.

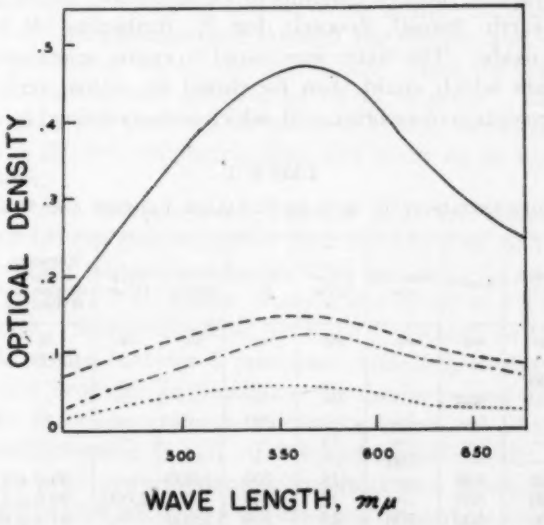

FIG. 4. Absorption curve for alkaline hydrolyzed calcium pantothenate, thiamine hydrochloride, ascorbic acid, and niacin. ——0.750 mg. calcium pantothenate. ——0.625 mg. thiamine hydrochloride. —·—·0.625 mg. ascorbic acid. ....3.125 mg. niacin.

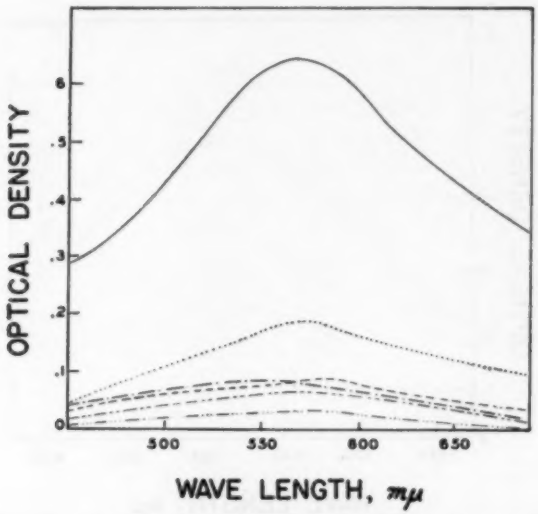

FIG. 5. Absorption curve for acid hydrolyzed calcium pantothenate etc. ——0.725 mg. calcium pantothenate. .....0.625 mg. pyridoxine hydrochloride. –·–·0.625 mg. thiamine hydrochloride. ——0.625 mg. ascorbic acid. – – – –3.125 mg. niacin. —·—·—3.125 mg. niacinamide.

In an attempt to separate interfering materials, solutions were chromatographed through columns of Decalso, talc, Lloyd's Reagent, infusorial earth, florosil, Zeocarb, Ion X, Amberlite IR 100H, and aluminum oxide. The latter was found to retain selectively calcium pantothenate which could then be eluted by either acid or alkali. With the exception of ascorbic acid, which is also retained by aluminum

TABLE I*

RECOVERIES OBTAINED ON ACID OR ALKALINE ELUTION AND HYDROLYSIS

Calcium Pantothenate	Thiamine HCl	Niacin	Ascorbic Acid	Riboflavin	Vitamin B_6	Corn Starch	Soya Flour	Recovery on Acid Elution and Hydrolysis	Recovery on Alkaline Elution and Hydrolysis
mg.	mg.	mg.	mg.	mg.	mg.	mg.	mg.	%	%
200	—	—	—	—	—	—	—	100.0	100.0
200	500	—	—	—	—	—	—	—	101.0± 2
200	—	500	—	—	—	—	—	—	100.0± 3
200	—	—	—	—	200	—	—	—	100.0± 3
200	—	—	—	25	—	—	—	—	100.0± 1
200	—	—	500	—	—	—	—	—	156.0± 6
200	500	500	—	25	200	2,000	—	99.0±3	100.0± 3
200	500	500	—	25	200	—	2,000	99.0±3	147.0±10
200	500	500	500	25	200	2,000	—	61.4±10	132.0±15
200	500	500	—	25	200	—	—	99.7±3	—
200	500	500	500	25	200	—	—	62.8±7	—
200	500	500	500	25	200	—	2,000	94.5±6	172.0± 8

oxide, the calcium pantothenate may thus be separated and subsequently determined.

Mixtures containing calcium pantothenate may be chromatographed and eluted with either acid or alkali and then may be hydrolyzed. However, if the mixture contained soya flour, it was found necessary to use acid elution and acid hydrolysis. In the first stages of this work, we used alkaline elution and hydrolysis. When a sample containing soya flour was encountered (Table I) abnormally high recoveries were obtained.

Procedure

Chromatographic Column. Use a glass column 12 mm. inside diameter, approximately 50 cm. in length and constricted at the lower end. Place a plug of glass wool in the lower end and slowly add 8 g. of special Chromatographic Grade Aluminum Oxide, applying gentle suction. Activate the column by washing with 50 ml. of 1:1 hydrochloric acid followed by 100 ml. of water immediately before use.

Preparation of Sample. Weigh an amount of sample containing about 200 mg. of calcium pantothenate into a 250 ml. volumetric flask and shake with 200 ml. of water for 10 minutes. Dilute to the mark and mix thoroughly. Filter or centrifuge this mixture and treat the clear solution as follows.

Chromatography. Pass an aliquot of the sample containing about 25 mg. of calcium pantothenate through the column and wash with successive portions of water, 100 ml., in all. Elute the column with 50 ml. of 1:1 sulfuric acid followed by 10 ml. of water. Reflux combined acid and water washings for one hour, cool to room temperature, transfer to a 250 ml. volumetric flask and make up to volume with distilled water.

Oxidation and Measurement. Transfer a 25 ml. aliquot of this solution to a 125 ml. glass-stoppered flask and add 5 ml. of 1:1 sulfuric acid. Adjust the temperature to 22–25°C. by placing in a water bath and add 5 ml. of 12% potassium bromide and 10 ml. of 5% potassium permanganate. Stopper the flask and leave in the bath for 10 minutes, then cool in an ice bath for 5 minutes. Add 20% freshly prepared sodium sulfite dropwise to decolorize the excess potassium permanganate. To the clear, colorless oxidized solution add 10 ml. of 2,4-dinitrophenylhydrazine (5 g./l. of 1:4 hydrochloric acid). Mix the solution with precipitate thoroughly and heat on a steam bath for at least 15 minutes after which cool the flask and contents to room temperature. Transfer the yellow precipitate to a small sintered glass filter, wash with five 5 ml. portions of water and dry for 30 minutes at 100°C. Meanwhile, drain the flask thoroughly and take up the last

traces of precipitate in hot pyridine using two successive portions of 3 ml. each. Transfer the pyridine washings to a 25 ml. volumetric flask. Fit the filter into a filtrator containing the 25 ml. volumetric flask. Add boiling pyridine in small portions to the filter, and gently triturate the contents with a glass rod. Apply suction, drawing the resultant pyridine solution through each time. Three or four washings are usually sufficient to dissolve and transfer all the precipitate to the volumetric flask. Make up the pyridine solution to exactly 25 ml.

Transfer a 5 ml. aliquot of this pyridine solution to a 100 ml. volumetric flask and add 50 ml. of water, followed by 5 ml. of 5 *N* sodium hydroxide. Dilute the solution to the mark and measure the absorbancy in a Beckman spectrophotometer set at 570 mu. This color is stable for about one hour.

Standard Curve

Purified calcium pantothenate (purified by the method of Levy, Weijlard and Stuler) (5) was used as the standard and carried through the procedure previously given.

In Table I may be seen the recovery of synthetic mixtures using both alkaline and acid elution and hydrolysis. Excellent results are obtained only in the absence of ascorbic acid.

In Table II results obtained by this chemical method on commercial samples are compared with results obtained by the presently accepted microbiological method. As may be seen, the results are in very good agreement.

TABLE II

COMPARISON OF COLORIMETRIC AND MICROBIOLOGICAL METHODS

Mixture	*Colorimetric*	*Microbiological*[1]
	mg./gm.	mg./gm.
Corn starch mixture of calcium pantothenate	72.6–72.0–70.2 Av. 71.0	76.9
Corn starch mixture of calcium pantothenate	73.7–75.5–74.5 Av. 74.7	79.7
Corn starch mixture of calcium pantothenate and vitamin B_2	73.7–76.6–78.4 Av. 76.2	70.6
Corn starch mixture of calcium pantothenate and vitamin B_2	77.7–78.4–74.5 Av. 76.9	77.5
Corn starch mixture of calcium pantothenate and vitamin B_2	74.5–73.7–75.5 Av. 74.6	73.6
Corn starch mixture of calcium pantothenate and niacinamide	73.7–75.5–74.5 Av. 74.6	76.2

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UNSATURATION OF CORN OIL—INHERITANCE AND MATURITY STUDIES [1]

B. BRIMHALL and G. F. SPRAGUE [2]

ABSTRACT

Iodine numbers of the oil were determined on High-oil and Low-oil corn inbreds, and their F_1, F_2, and backcross populations. The mean of the F_2 was midway between the parental means. Inheritance of iodine number in this cross is probably conditioned by a small number of genes, in the order of four as a minimum.

A highly significant negative correlation was found between iodine number and oil content of the germ, and between iodine number and oil content of the kernel. The latter relationship was confirmed in an unrelated stiff-stalk synthetic variety. However, the relationship was not so close as to preclude the development of high oil strains with medium or high iodine number. The correlation between iodine number and weight per germ was not significant.

Ears from two inbreds and a single cross were harvested at different stages of maturity beginning at 20 days after pollination. Iodine number of the oil remained fairly constant throughout the period studied. Data obtained at five-day intervals during the growing period showed that weight per kernel and weight per germ continued to increase until 50 days after pollination. The per cent of oil in the kernel was essentially constant after 35 days except for the High-oil strain which was still increasing at 60 days. The per cent of oil in the germ increased to a maximum at about 30 days after pollination and then decreased with maturity by as much as 25%.

Measurements of unsaturation in corn oil (iodine number) have been carried out on composite oil samples obtained as by-products of the milling process, but there is apparently no literature dealing with the variation which may be expected among different ears or different lines of corn. Extensive studies of this type have been conducted with

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[2] Research Associate, Iowa Agr. Exp. Sta., and Senior Agronomist, Bureau of Plant Industry, Soils and Agricultural Engineering, respectively.

other oil-bearing seeds, notably flax, soybeans and cottonseeds. With flax and soybeans it has been possible to breed varieties with iodine numbers which make their oils more suited for specific uses. For example, soybean oil with a high iodine number is used in paints; that with a lower iodine number is used in salad oils. Similar attempts to improve the quality of corn oil have not been made, probably because the oil comprises a relatively low percentage of the kernel (about 4.0%) and its iodine value is already suited for the edible oil trade. However, in conjunction with the recent breeding program at the Iowa Agricultural Experiment Station to increase the quantity of oil in commercial corn hybrids (6, 7), it seemed desirable to investigate the possibilities for variation in iodine number.

Materials and Methods

The High-oil and Low-oil inbreds were two-generation selfs isolated from Illinois High-oil and Low-oil strains. The inbreds, F_1, F_2, and backcross generations were all grown in the same plot in 1947. After harvesting, the ears were dried in an artificial drier at approximately 100°F., then shelled. Each sample represents the grain from a single ear of corn.

All samples were ground 20-mesh in an intermediate model Wiley mill. For oil analysis, two grams of ground corn was placed in the cup of a Bailey-Walker flask and extracted 12 hours with 25 ml. of petroleum ether (Skelly A; b.p. 28–38°C.), after which the ether extract was transferred to weighed beakers, evaporated, and the weight of oil obtained. All values are expressed on a dry weight basis.

Iodine absorption numbers were determined by the Hanus method (1). For separation of the germ, the kernels were soaked in warm water and hand-dissected.

Results

Inheritance of Iodine Number. Iodine number was determined on oil samples from the High-oil (HO) and Low-oil (LO) inbreds, and their F_1, F_2, and backcross populations. This cross was chosen because it represents the greatest available range in oil content (6). The frequency distributions for each population and the means are shown in Table I. It was necessary to use a composite sample from the Low-oil inbred since sufficient oil was not available from a single ear.

As is frequently the case with quantitative characters, the use of Powers' formulae (5) does not allow a conclusion as to the type of inheritance involved because the observed mean iodine numbers of the F_2 and backcross populations are in good agreement with means calculated either for arithmetic or for geometric gene action. If we as-

TABLE I

FREQUENCY DISTRIBUTIONS OF IODINE NUMBER AND OF OIL PERCENTAGE FOR SAMPLES OF CORN REPRESENTING SIX GENETIC POPULATIONS

Parentage	Class Midpoints of Iodine Number (Hanus)													Mean Iodine Number
	110.5	112.5	114.5	116.5	118.5	120.5	122.5	124.5	126.5	128.5	130.5	132.5	134.5	
LO													10[1]	135[1]
HO	4	2	7	1										113
(HO×LO)F_1						6	7	6						123
(HO×LO)F_2				6	10	23	33	23	24	9	5	5	1	124
(HO×LO)×HO	2	6	14	18	12	12	2	1						117
(HO×LO)×LO						3	11	14	11	13	9	3	3	127

[1] Composite of ten samples.

Parentage	Class Midpoints of Oil Percentage																			Mean Oil Percentage
	0.75	1.35	1.95	2.55	3.15	3.75	4.35	4.95	5.55	6.15	6.75	7.35	7.95	8.55	9.15	9.75	10.35	10.95	11.55	12.15	12.75	
LO	3	15	4																			1.41
HO																	1	8	5	3	2	11.42
(HO×LO)F_1							10	10														4.70
(HO×LO)F_2				3	12	27	36	39	20	8	1											4.55
(HO×LO)×HO									5	6	19	21	7	12	3							7.54
(HO×LO)×LO			5	34	22	7																2.50

sume that inheritance is arithmetic and that genetic extremes in iodine number of the oil have been reached, application of the Castle-Wright formula (2) indicates that iodine number is controlled by a minimum of four genes. However, the limitations which have been previously discussed (6) in applying this formula to data on percent of oil in the kernel are equally important here.

There is a highly significant negative correlation between iodine number and per cent of oil in the germ, both among the six populations of the cross (Fig. 1) and within the three segregating populations (Table II). For the F_2 samples, a high negative correlation was also

TABLE II

RELATIONSHIP OF IODINE NUMBER TO OIL CONTENT AND WEIGHT OF GERM

Type of Population	Correlation Coefficient (r) of Iodine Number with		
	% Oil in Germ	% Oil in Kernel	Weight per Germ
F_2	−0.563[2] (99)[1]	−0.405[2] (139)	−0.114 (99)
F_1×HO	−0.405[2] (67)		
F_1×LO	−0.348[2] (51)		
Stiff-Stalk Synthetic		−0.571[2] (170)	

[1] Numbers in parentheses are numbers of samples analyzed.
[2] Significant at the 1% level.

found between iodine number and per cent of oil in the kernel. This relationship was confirmed with an unrelated variety known as Stiff-Stalk Synthetic which was segregated for oil content (range, 3.9–8.0% oil; mean, 5.2%).

Effect of Maturity and Season. Although limited in scope, the results with four corn inbreds at maturity (Table III) show remarkably good agreement among iodine numbers for three successive seasons.

Maturity studies were carried out on the High-oil inbred, an inbred Iowa I 198, and a single cross I 198×Hy. These were grown in 1948 and representative ears harvested at five-day intervals beginning with 20 days after pollination. The results, presented in Table IV, show that iodine number remains fairly constant from 25 days after pollination of the corn, and in some cases is constant after 20 days.

Several other characteristics of the maturing kernel were followed on the same samples and recorded in Table IV. The per cent of oil in the germ increased to a maximum at about 30 days after pollination

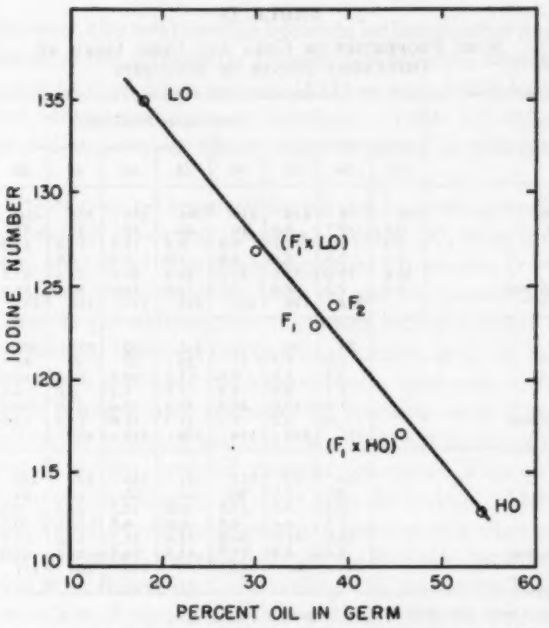

FIG. 1. Relationship between mean iodine number and mean oil content of germ in six populations of a cross between high- and low-oil inbreds.

TABLE III

EFFECT OF SEASON ON IODINE NUMBER OF OIL FROM CORN INBREDS

Inbred	Year Grown	Iodine Number of Oil	Oil in Corn
			%
B-5	1946	116	
	1947	115	3.0
	1948	112	
B-12	1946	128	
	1947	129	3.4
	1948	125	
B-8	1946	115	
	1947	113	4.4
	1948	117	
B-2	1946	122	
	1947	123	4.9
	1948	125	
Commercial corn oil		123	

TABLE IV

SOME PROPERTIES OF CORN AND CORN GERM AT DIFFERENT STAGES OF MATURITY

	Days after Pollination									
	15	20	25	30	35	40	45	50	55	60
F_1 (I 198×Hy)										
Wt./kernel, mg.	34	79	135	210[1]	264	316	335	400	402	405
Wt./germ, mg.	0.7	2.7	8.6	19	26	36	43	52	54	55
% germ	2.0	3.4	5.7	9.0	9.8	11.5	12.8	13.0	13.4	13.5
% oil in corn	2.6	2.9	3.4	3.9	4.0	4.6	4.0	4.6	4.3	4.1
% oil in germ	16.8	25.0	33.8	33.0	33.8	31.0	29.3	27.7	27.3	25.4
Wt. oil in corn, mg.	0.9	2.3	4.6	8.2	10.6	14.5	13.4	18.4	17.3	16.6
Iodine No.	—	112	118	117	118	118	116	117	120	117
I 198										
Wt./kernel		53	94	176	221	250	271	302	312	309
Wt./germ		1.1	4.5	17	26	27	39	44	44	44
% germ		2.2	4.8	9.6	11.7	10.8	14.4	14.5	14.1	14.2
% oil in corn		3.1	3.4	4.4	5.1	4.4	4.6	4.6	4.6	5.2
% oil in germ		27.8	32.6	34.4	34.5	32.6	27.0	26.0	26.9	26.7
Wt. oil in corn, mg.		1.6	3.2	7.7	11.3	11.0	12.5	13.9	14.4	16.1
Iodine No.		117	119	114	116	118	115			
High oil										
Wt./kernel		49	77	117	131	189	197	229	208	206
Wt./germ		3.1	7.1	16	26	35	42	47	48	48
% germ		6.3	9.2	13.9	19.6	18.5	21.3	20.5	23.0	23.2
% oil in corn		4.9	6.4	9.8	8.5	8.8	11.0	10.5	11.9	12.5
% oil in germ		38.8	41.8	50.0	51.0	51.2	51.0	47.2	47.6	48.6
Wt. oil in corn, mg.		2.4	4.9	11.5	11.1	16.6	21.7	24.0	24.8	25.8
Iodine No.		106	106	112	—	110	105	111		

[1] The F_1 ears were picked at 32 days instead of 30.

and then decreased with maturity by as much as 25%. This trend was more pronounced with I 198 and the single cross than with the High-oil inbred.

The weight per kernel and weight per germ continued to increase until 50 days after pollination, and per cent of germ in the kernel increased until at least 45 days. However, the per cent of oil in the kernel was essentially constant after 35 days, except for the High-oil inbred which showed a tendency to increase in this respect even at 60 days. The total weight of oil increased to a maximum at the 60 day harvest period for the two inbreds but at 50 days for the F_1 hybrid.

Discussion

Attempts to modify the characteristics of corn oil by breeding procedures have been almost completely lacking. Since it now appears feasible to produce commercial hybrids with at least twice the oil content of those commonly grown, the question arose as to whether the resulting change in the quality of the oil was great enough to effect its present uses or perhaps to extend them. The results presented in Table II indicate that in breeding for increased oil percentage some reduction in iodine number of the oil produced will amost certainly

result. However, the relationship between iodine number and per cent oil in the kernel is not sufficiently close to preclude the development of high oil strains having either normal (123) or high (130) iodine values if a sufficient commercial demand develops. Table III illustrates the variation in iodine number which may be found in different imbred lines.

The results of corn oil reported herein are in good agreement with those on soy bean oil in several respects. Weber (8), in an interspecific soybean cross, found (a) significant negative correlation ($r = -0.311$) between the iodine numbers of soybean oil and the percentage of oil in the seed of the F_2 population, (b) the mean iodine number of the F_2 was exactly midway between the parental means, and (c) inheritance of iodine number is probably determined by a minimum of five genes.

Lehberg, McGregor and Geddes (4), working with flaxseed harvested at successive stages of maturity, showed that moisture decreased and dry kernel weight and oil content increased with progressive maturity up to approximately 30 days after flowering. Although unsaturation of the oil developed somewhat more slowly than oil deposition, the iodine number increased by 60 to 80 units between the 6th and 30th days after flowering. According to a review by Hilditch (3), the iodine numbers of cottonseed and soybean oils, as well as linseed, increased markedly with maturity. The data in Table IV indicate that the unsaturation of corn oil changes little, if any, between 20 days after pollination and maturity. The effects of season and climate on the quality of corn oil have not been adequately investigated. These factors have a pronounced effect on unsaturation of soybean and linseed oils.

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DEVELOPMENT OF FREE FATTY ACIDS DURING STORAGE OF BROWN (HUSKED) RICE [1]

I. R. HUNTER, D. F. HOUSTON, AND E. B. KESTER

ABSTRACT

Development of free fatty acid in commercially dehulled Caloro brown rice stored 22 weeks at moisture levels of 3.9, 6.6, 9.5, 11.8, and 14.1% (wet basis) and at temperatures of 0° to 2°, 25°, and 35°C. (32° to 35.5°, 77° and 95°F.) increased with the moisture content and temperature of storage. An increase in free fatty acids of approximately 1% per month resulted at 0°C. (32°F.) in rice containing 14.1% moisture, and at 25°C. (77°F). in rice with 6.6% moisture. At 25°C. (77°F.), the initial rates of acid formation were reduced about four-fold by lowering the moisture content from 14.1 to 9.5% and sixteen-fold by drying to 3.8%. Two portions of a separate lot of Caloro brown rice, prepared in the laboratory from paddy rice stored 7 weeks at 1.5°C. (35°F.) with 21.9% moisture, were dried to 13.9% and 15.2% moisture and stored at 25°C. (77°F.). Development of free fatty acids was generally similar to that of the first lot. Observed differences indicate possible variations in a single variety of rice under differing cultural, storage, and milling conditions. Other varieties would be expected to give similar results.

The development of free fatty acids in grains during storage has been included in cereal and oil-seed deterioration studies by numerous investigators (4, 6, 8–10, 13, 15, 16–22, 25, 26, 29–32) during the past twenty-five years, and the measurement of fat acidity has been recommended (29, 30) as a criterion of the commercial condition of the grain. Fatty acids are readily formed by lipases from fats in grain, particularly under storage conditions of high temperature and moisture content (9, 10).

Geddes and co-workers (4, 8, 9, 16–21), Gilman and Semeniuk (10), and Nagel and Semeniuk (22) have evaluated some effects of mold growth in the deterioration of corn, cottonseed, soybeans, and wheat. Molds such as *Aspergillus* and *Penicillium* which produce lipases (8) will grow on grain with moisture contents in equilibrium with air of about 75% relative humidity or above. This relative humidity corresponds with 14 to 15% moisture (wet basis) for rice (12). At relative humidities below 75%, the increases in fat acidity are more likely attributable to causes other than microorganisms,

[1] Manuscript received October 6, 1950. Contribution from Western Regional Research Laboratory, Bureau of Agricultural and Industrial Chemistry, Agricultural Research Administration, U. S. Department of Agriculture, Albany, California. Report of a study made under the Research and Marketing Act of 1946.

although damaged, broken and dead seeds are reported (10, 16) to be more readily attacked than sound ones because of greater availability of nutrients.

The role of inherent seed enzymes in these processes is not so clearly understood (8, 9, 19). In this connection, Kyame and Altschul (15) have described the differences in free acid formation between immature cottonseed with its active enzymes and the dormant mature seed, which reportedly has no lipase activity (23). Differences have also been pointed out (27) in the reactions of rice and wheat to test environments.

Brown or husked[2] rice deteriorates in quality during storage, chiefly because the approximately 2% of oil present is subject both to oxidative rancidification and lipolytic formation of free fatty acids. The bran layers are bruised and scarified to varying extents during dehulling processes (14, 24). Exposure of a portion of the oil to the atmosphere results, as well as intimate mixing with other components, including enzymes. The rate of deterioration of the oil might be expected to vary, accordingly, from one lot of rice to another as a result of variation in milling processes, aside from any cultural or varietal differences.

Lipolysis, and the formation of free fatty acids, in rice has been shown to be largely enzymatic in nature (5, 25, 28), although the origin of the lipolytic enzyme has not been definitely established. Reduction of moisture content or storage temperature, or both, should reduce the rate of enzymatic activity from whatever source and permit prolonged storage of brown rice.

The effects of differing storage temperatures and moisture levels on rate of formation of free fatty acids in a representative sample of California-grown Caloro brown rice are reported herein. Also presented for comparison are the storage characteristics at 25°C. (77°F.) of two portions of brown rice (prepared at 14 and 15% moisture levels for other studies) from a lot having an entirely different history.

Materials and Methods

The brown rice used for the main series of storage tests was commercially dried and milled from paddy of 1948 harvest and contained 14.1% moisture. Lots weighing 2 kg. each were dried at 38°C. (100°F.) in a tunnel drier to approximately 12 and 10% moisture. Two additional 2-kg. lots were similarly dried to 10%, and then to

[2] Husked rice is the term proposed as standardized International Rice Terminology by the International Rice Commission of the Food and Agricultural Organization of the United Nations. In the Report of the First Session of the International Rice Commission (11), it is stated on p. 41 that husked rice is "Rice from which the husk only has been removed; it still retains the bran layers and most of the germ." The comment is added that it is "commonly referred to as 'brown rice' even though there are varieties having red or white bran coats."

7 and 4% moisture, respectively, by vacuum desiccation over phosphorus pentoxide at room temperature. All samples were held at 0°C. (32°F.) until storage tests were started. Each sample was subdivided and the subsamples were stored in tightly closed screw-cap jars at temperatures of 0° to 2°C. (32° to 35.5°F.), 25°C. (77°F.), and 35°C. (95°F.). Individual jars were removed at intervals as needed for analytical determinations.

In addition, two portions of brown rice were prepared by dehulling, with laboratory shelling stones, portions of newly dried paddy from the 1948 harvest, which had been stored seven weeks at 1.5°C. (35°F.) with the original 21.9% field moisture. These samples, with 13.9 and 15.2% moisture, originally prepared for milling tests at 14 and 15% levels, were stored at 25°C. (77°F.).

Moisture contents of all samples were determined by measuring the loss in weight on heating 20-g. samples in a forced-draft oven at 104–106°C. (220–225°F.) for 16 hours, and calculating the results on a wet basis. This is a slight modification of the official method of the American Oil Chemists' Society (2) and the Association of Official Agricultural Chemists (3) for moisture in cottonseed used by Christensen and Gordon (7) with wheat and corn and by Karon and Adams (12) in the determination of hygroscopic equilibrium of rice.

Determination of free fatty acids was made by an adaptation of the colorimetric titration of Ames and Licata (1) as used in the Physicochemical and Analytical Division of this Laboratory. The rice was ground in an intermediate Wiley mill to pass a 20-mesh sieve. An amount calculated to give a 3- to 10-ml. titration with 0.01 *N* alcoholic potassium hydroxide was weighed into a tared Whatman extraction thimble, and extracted 5 hours with 100ml. of absolute ether into a 125-ml. flask. The ether was removed on a steam bath, finally in a stream of nitrogen. To the residual oil were added 10 ml. of the isopropanol-benzene solvent and 6 drops of the phenolphthalein indicator. Titration was performed with 0.01 *N* potassium hydroxide in isopropanol freshly diluted from an accurately checked stock solution about 0.1 *N*. Extractions were made on several thimbles as for the acid determination, and the average titration of the resulting extract residues was used as a blank to correct the acidity values. The total oil content of the sample was separately determined by a 16-hour ether extraction. The percentage of free fatty acids (as oleic) was calculated as follows:

$$\% \text{ free fatty acid in oil} = \frac{A \times \text{normality of KOH} \times 2820}{B \times C},$$

where A is the titration in ml. for the sample minus the correction in ml. for the thimble blank titration, B is the weight of the sample in grams, and C is the percent of oil in the sample. The reported values are averages from at least two determinations.

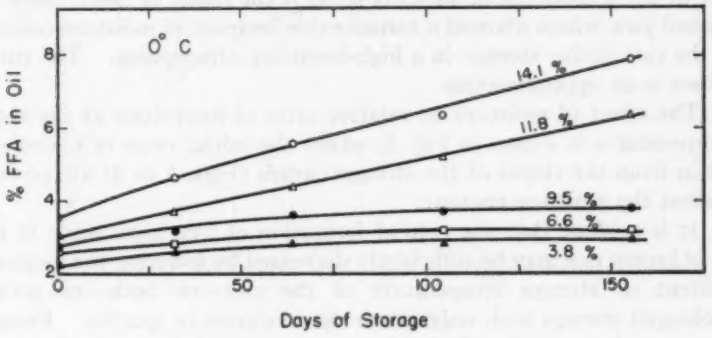

FIG. 1. Development of free fatty acids in commercial brown rice of different moisture contents stored at 0°C. (32°F.).

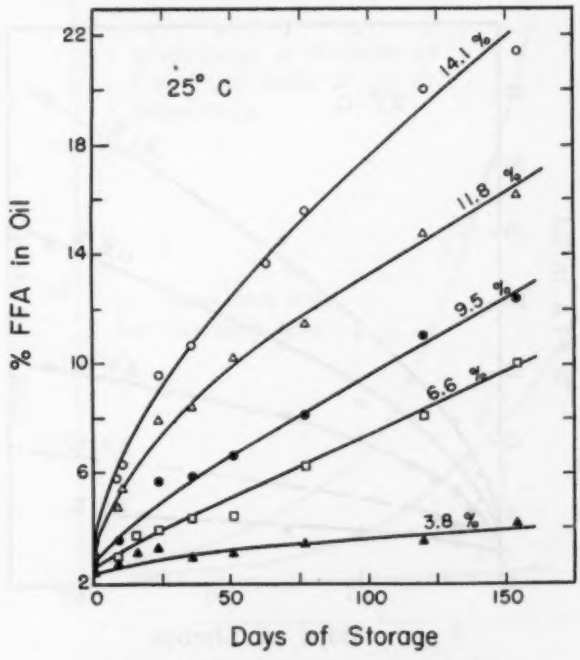

FIG. 2. Development of free fatty acids in commercial brown rice of different moisture contents stored at 25°C. (77°F.).

Results and Discussion

The percentages of free fatty acid in the oil of the brown rice after differing periods of storage at the several conditions are presented graphically in Figs. 1 to 4. The lack of values for acid percentages in rice of 6.6% moisture at 35°C. (Fig. 4) is the result of faulty seals on several jars, which allowed a considerable increase in moisture content of the rice during storage in a high-humidity atmosphere. The curve shown is an approximation.

The effect of moisture on relative rates of hydrolysis at the three temperatures is shown in Fig. 5, where the initial rates of hydrolysis taken from the slopes of the storage curves (Figs. 1 to 4) are plotted against the moisture content.

It is evident that the rate of formation of free fatty acids in the oil of brown rice may be sufficiently decreased by lowering the moisture content or storage temperature of the rice—or both—to permit prolonged storage with only minor deterioration in quality. From a commercial standpoint, low-temperature storage would be the more feasible as it would avoid considerable weight loss, the difficulty

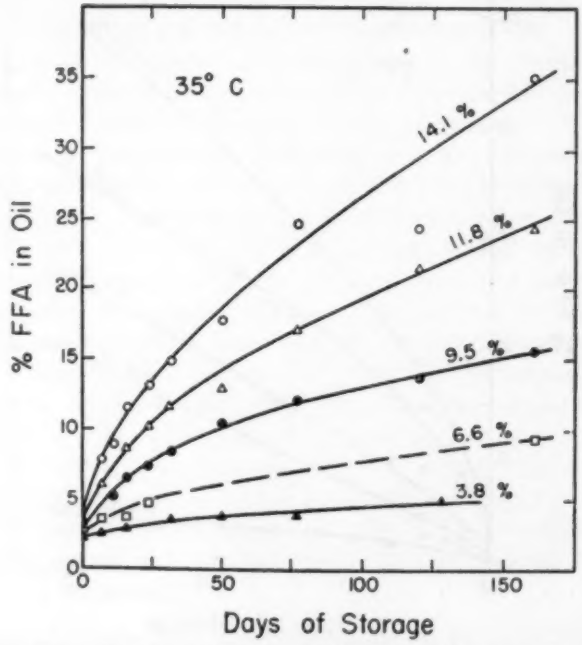

FIG. 3. Development of free fatty acids in commercial brown rice of different moisture contents stored at 35°C. (95°F.).

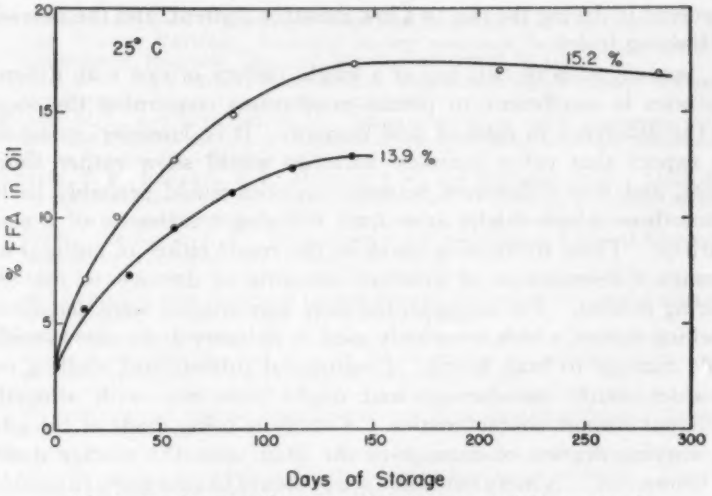

FIG. 4. Development of free fatty acids in laboratory-hulled brown rice of different moisture contents stored at 25°C. (77°F.).

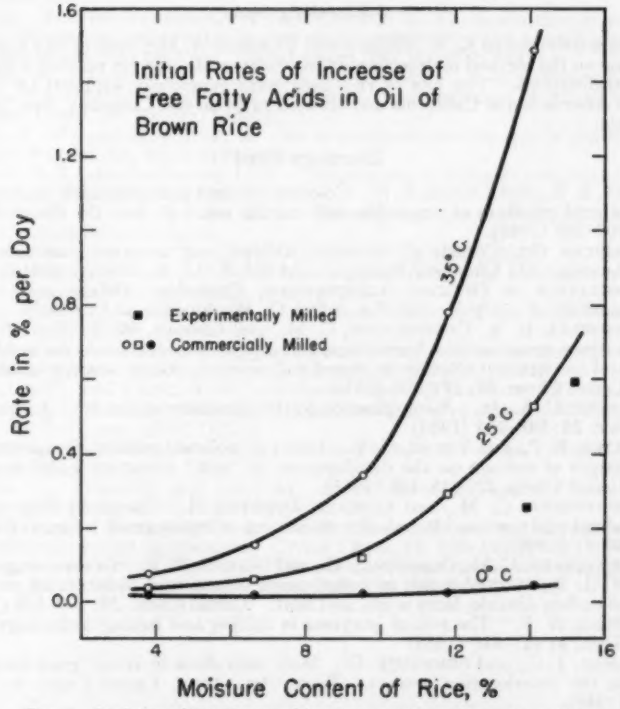

FIG. 5. Variation of initial rates of free fatty acid formation with moisture content of brown rice at three temperatures.

involved in drying the rice to a low moisture content, and the necessity of keeping it dry.

Investigation of two lots of a single variety of rice with different histories is insufficient to permit conclusions concerning the source of the difference in rates of acid increase. It is, however, reasonable to expect that other common varieties would show rather similar rates, and that differences between varieties would probably be less than those which might arise from differing treatments of a single variety. These differences could be the result either of cultural and storage differences or of different amounts of damage to the bran during milling. For example, the rices here studied were dehulled in shelling stones, which are widely used in industry and cause considerable damage to bran layers. Commercial rubber-lined shelling rolls produce much less damage and might yield rice with altogether different storage characteristics. A study is being made of the effect of varying degrees of damage to the bran upon the storage quality of brown rice. A more extensive study would be necessary to establish general limits for rates of free fatty acid development.

Acknowledgments

We are indebted to K. T. Williams and Elizabeth A. McComb of this Laboratory for advice on the method of determining free fatty acids, and for making a number of the determinations. The lots of rice used were generously supplied by the Rice Growers Association of California and Rosenberg Bros. and Company, San Francisco, California.

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AIR BUBBLES IN MACARONI DOUGHS[1]

G. N. IRVINE and J. A. ANDERSON

ABSTRACT

Studies on mixing macaroni doughs in oxygen, air and nitrogen indicate that the solubility of the gas in the dough has a marked effect on the number of bubbles in the finished product. At the pressures used in macaroni processing, all the occluded air bubbles dissolve in the dough. When the pressure is released, the gas tends to precipitate out on small nuclei in the dough; the amount of pressure and time of application appear to influence the number of these nuclei in the dough. Examination of pressed doughs under the microscope indicates that the bubbles apparently "move" through the dough in the direction of highest water concentration. Hence with macaroni, gas bubbles can escape from the dough by migrating to the central hollow in the tube; slower drying will facilitate this escape.

A high degree of translucency has traditionally been considered one of the necessary attributes of high quality macaroni products. The first quantitative work on translucency (or opacity) of macaroni was done by Cunningham and Anderson (1) who devised an instrument for measuring the optical opacity coefficient for macaroni discs made by a modification of the original micro disc procedure of Fifield *et al.* (2). Effects of various processing factors on the opacity of the discs were recorded. The mixing time, and time and degree of pressure applied to the dough in processing, were found to have the most marked effects on opacity, which appears to be largely a function of the number and size of the air bubbles in the finished product. Later, Smith *et al.* (5) studied microscopically the variations in size and number of bubbles resulting from various pressures and pressing times on micro discs. Their work shows that high translucency is associated with small numbers of large bubbles in the macaroni, resulting from high pressures and long pressing times, while low translucency is associated with large numbers of small bubbles, resulting from low pressures and short pressing times. Sibbit and Harris (4) showed that the color characteristics of the samples obtained by Smith *et al.* varied widely with the number and size of the air bubbles and state that translucency measurements alone give only a fair indication of macaroni color.

In attempting to account for the results of their investigations, both Cunningham and Anderson and Smith *et al.* suggest that pressure

[1] Manuscript received September 18, 1950. Paper No. 109 of the Grain Research Laboratory, Board of Grain Commissioners for Canada, Winnipeg, Manitoba, and No. 282 of the Associate Committee on Grain Research (Canada).

causes the small bubbles introduced into the dough during mixing to coalesce into larger bubbles. Cunningham and Anderson, however, state that this hypothesis is not adequate to account for all the observations of their experiments.

Some recent observations made in this laboratory in connection with another study suggested a new hypothesis concerning the formation of air bubbles in macaroni products, and several follow-up experiments were made to elaborate the hypothesis.

During an experiment in which macaroni doughs were mixed in nitrogen, air, or oxygen, samples of the doughs were taken after a short mixing period, shaped by hand into flat cakes, and allowed to dry in a dessicator. Two interesting phenomena were observed: the color of the dried cakes was very markedly different, varying from a clear deep yellow for the sample mixed in oxygen to almost dead white for the sample mixed in nitrogen; and, on breaking the cakes open, it was found that the center of each cake was honeycombed with gas pockets which were largest in the center of the cake.

This experiment was then repeated using the disc method of Cunningham and Anderson in which the discs are pressed for seven minutes at 1000 lb. per sq. in. Again the color differences were marked although to a lesser degree than for the samples prepared without pressing. The honeycomb effect was not as noticeable, but an indication that it did occur to a small extent was obtained by examining the discs under the microscope.

Since the loss of pigment during mixing is greatest in oxygen and least in nitrogen, the yellow color of the discs (greatest for oxygen, and least for nitrogen) are in the opposite order to pigment content.

In the experiment in which no pressure was applied, the smaller number of gas bubbles in the oxygen mixed sample could not be accounted for by coalescence under pressure. However, the results can be explained, in some measure, in terms of the solubility of the three gases in the dough. Oxygen is the most soluble of the three gases and hence more of the gas occluded by the dough during mixing will dissolve. As the cake dries, some of the oxygen present in the form of small bubbles, probably dissolves in the water phase and escapes as the water evaporates at the dough surface. As drying proceeds and the outer surfaces harden, the bulk of the small bubbles of oxygen, air, or nitrogen, representing a high aggregate surface energy, can only reduce this energy by going into solution and precipitating on other bubbles nearer the center of the dough, which will still be plastic. Thus, with further drying, the outer layers become almost completely devoid of bubbles, and bubbles in the center of the disc become much larger. The extent to which this phenomenon occurs will be a function of the solubility of the occluded gas.

If an estimate of the amount of gas occluded by the dough during mixing is made, knowing the volume of water in the dough, it becomes evident that at 1000 lb. per sq. in. pressure all the gas occluded by the dough should dissolve. That this occurs was confirmed by subjecting a macaroni dough to pressure in a clear plastic cylinder. At a pressure just over 600 lb. per sq. in. for the sample used, the dough suddenly changes from an opaque putty white color to a translucent yellow, and it seems extremely probable that this is associated with the gas bubbles going into solution in the dough. When the pressure is released the dough will be supersaturated with gas to a degree depending on the solubility of the gas.

As the dough rests, the gas will tend to precipitate out from the dough. The ease with which this can occur will depend on the number of nuclei present in the dough at which the gas bubbles may reform. These nuclei are probably tiny gas bubbles which do not completely dissolve under pressure, and the number of these will decrease with higher pressures and longer pressing times. The importance of these nuclei was demonstrated by the following experiment: a micro disc was prepared in the usual way sandwiched between celluloid discs which had been scratched on the inside surface. On removal of the sandwich from the press, examination under the microscope revealed tiny gas bubbles along the scratched pattern. In a matter of hours, the bubbles greatly increased in size, indicating that the gas was coming out of solution at these centers.

A further experiment confirmed the ability of the small gas bubbles formed at nuclei in the dough to "migrate" towards the center of highest water concentration. A macaroni disc was prepared with unscratched celluloid discs and one of the celluloid discs was then removed. The sample was examined periodically under the microscope for two days. As drying progressed on the open side of the sandwich, the bubbles on the celluloid side became larger and also "moved" nearer to the celluloid disc, where the water concentration was highest. At the end of two days, very large gas bubbles had formed in the dough very close to the dough-celluloid interface. It is probable that the bubbles do not actually move through the dough. The large surface energy possessed by a large number of small bubbles can be reduced by transport of gas in solution from the small bubbles to the larger bubbles in much the same way as a small drop of water will "distill" in a saturated atmosphere on to a larger drop. This process will occur most readily where the internal pressure of the dough on the larger bubbles is least, that is in the area of highest water concentration. This "movement" of the bubbles probably accounts for the often observed fact that experimental scale macaroni contains more gas bubbles than the

commercial product. The commercial product is usually dried much more slowly and hence there is a greater opportunity for the gas bubbles to escape into the open center tube of the macaroni, which is the area of highest water concentration.

It is necessary now to account for observations on the numbers and sizes of bubbles resulting from various processing conditions in terms of these new facts, namely: most of the air goes into solution in the dough under pressure; bubbles reform from the supersaturated solution at active centers or nuclei in the dough when the pressure is released; these bubbles can apparently migrate towards an area of the dough which has the highest water content.

According to Cunningham and Anderson (1), processing conditions which had the most marked effect in improving translucency were high absorption, high pressure, long pressing time, increased mixing temperature, and decreasing mixing time. Smith *et al.* (5) agree that, with all other conditions constant, high pressures and long pressing times result in fewer and larger bubbles. A short mixing time obviously reduces the number of air bubbles occluded by the dough. Higher absorption increases the amount of gas dissolved in the dough and decreases the internal pressure of the dough; both effects should reduce the number of bubbles. Higher pressure and longer pressing time will both result in fewer nuclei in the dough and lead to the formation of fewer and larger bubbles. Higher mixing temperatures will decrease the solubility of the gas in the dough, but greatly decrease its viscosity; hence, the greatly reduced internal pressure more than offsets the decrease in solubility.

There is one further observation which may assist in accounting for the observed behavior of the gas bubbles. Martin *et al.* (3) showed that increasing absorption, higher pressures and longer pressing times reduce the viscosity of doughs during macaroni processing. This reduced viscosity represents a reduction in the internal pressure of the dough and hence would facilitate the formation of larger and fewer bubbles of gas in the dough.

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COMMUNICATION TO THE EDITOR

Turbidity Developed in Sodium Hydroxide Suspensions of Flour upon Acidification

DEAR SIR:

In 1941 Zeleny[1] suggested using a buffered system of pH 7.8 to develop maximum turbidity in a neutralized alkaline extract of wheat flour. It has since been found that very sharp and narrow turbidity maxima can be obtained in unbuffered sodium hydroxide suspensions of wheat flour upon acidification at somewhat lower pH values. When 1 ml. of the centrifugate of a suspension of 1 g. of flour in 50 ml. of 0.05 *N* sodium hydroxide solution, diluted with

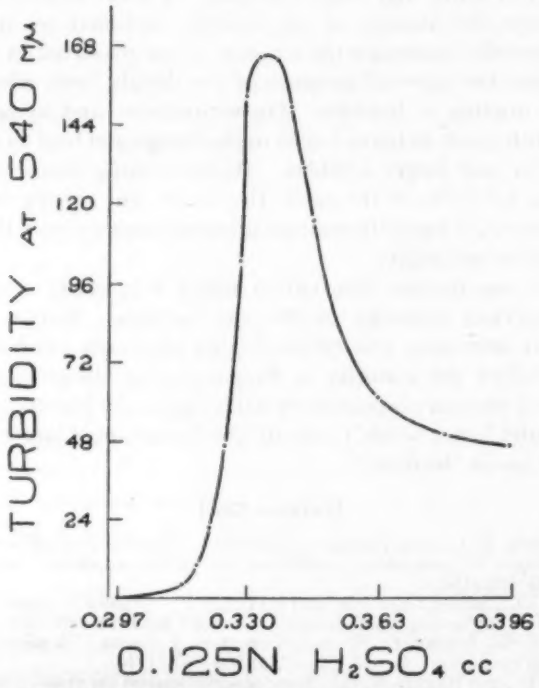

FIG. 1. Effect on turbidity of acidification of alkaline flour extracts. The maximum turbidity occurs upon acidification to about pH 5.75.

[1] Zeleny, L. A simple photometric method for determining the protein content of wheat flour. Cereal Chem. 18: 86 (1941).

5 ml. of water in a Klett-Sumerson photometer tube, is gradually neutralized with 0.125 *N* sulfuric acid solution, a sharp maximum turbidity develops at a pH of about 5.75. Moreover the distance between inflection points of the plot of turbidity against acid additions was equal, in most cases, to less than 0.02 ml., indicating the narrowness of the range in pH over which the most noticeable effects of gluten swelling, hydration, or water imbibition on turbidity are influenced by hydrogen ion concentrations. See Fig. 1.

A correlation coefficient of 0.955 for the relation between maximum turbidity as developed with dilute sulfuric acid and flour protein determined by the Kjeldahl method was obtained for a series of 20 flours with protein contents from 6.0 to 12.5%. Unfortunately loaf volume figures for these flours are not available. While there is little to recommend them as substitutes for Kjeldahl determinations, it is felt that these curves may be of interest in the characterization of gluten behavior, or may find application in Zeleny's original method, in his sedimentation technic[2] or that of Berliner and Koopman[3], or in Finney and Yamazaki's[4] water retention procedure.

R. J. BUTLER,
Capitol Milling Company,
Los Angeles, California

January 8, 1951

[2] Zeleny, L. A simple sedimentation test for estimating the bread-baking and gluten qualities of wheat flour. Cereal Chem. 24: 465 (1947).
[3] American Association of Cereal Chemists. Cereal Laboratory Methods. 3rd Ed., p. 66. Am. Assoc. Cereal Chem.: Omaha, Nebr. (1935).
[4] Finney, K. F., and Yamazaki, W. T. Water retention capacity as an index of the loaf volume potentialities and protein quality of hard red winter wheats. Cereal Chem. 23: 416 (1946).

BOOK REVIEW

Lebensmitteltechnologie: Einfuhrung in die Verfahrenstechnik der Lebensmittelverarbeitung. By Rudolf Heiss. Edited by J. F. Bergmann, Munich (Germany), 323 pp. Price: DM 27.60.

The subject matter of this book is divided into two main parts. The first one (p. 1–82), which gives a short review of the fundamental operations used in food processing, is divided into four sections:

1. Operations leading to changes in the density of matter: crushing, grinding, pressing.
2. Mixing: agitating, kneading, emulsifying.
3. Various techniques of separation: (a) Mechanical separations: extraction, size separation, centrifugation, filtration; (b) Thermal separations: evaporation, crystallization, drying, etc.
4. Other techniques used in food industry: ion exchange, conveying, sterilization, etc.

In the second part (p. 83–337), the author describes briefly the manufacture of a number of food products. These descriptions are arranged according to the classifica-

tion given in the first part. For example, under mixing and emulsifying, the author describes among other things the manufacture of margarine, chocolate, and alimentary pastes. Most of the important food industries are dealt with in this part. Among those discussed are the industries involving the manufacture or processing of cereals, animal and vegetable fats and oils, dairy products, starch, chocolate, coffee substitutes, fruits, meat, etc. Finally there is a special section dealing with industries using microorganisms, such as those involving the manufacture of beer, cheese, wood sugar, acetic acid, citric acid, lactic acid, etc. This book contains 223 helpful illustrations as well as a complete subject index. The author does not claim to give a thorough treatment of the subject, but says that his aim is to give the technician a survey of the possibilities of the food industry and of the main steps involved in food processing. Consequently none of the subjects is treated in detail, but what is given is well presented, well organized and the important points are emphasized.

This book will be of value to the food technologist as a source of general information, and since it presents a clear survey of the field it will be of special interest to the student in food technology.

LOUIS S. CUENDET
Division of Agricultural Biochemistry
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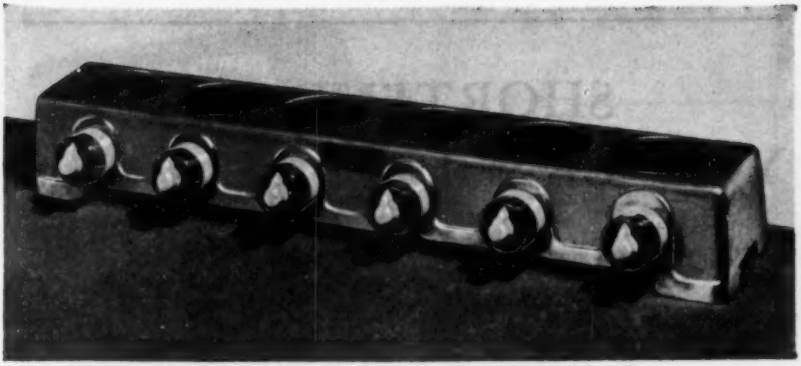

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Laboratory Apparatus • Chemicals

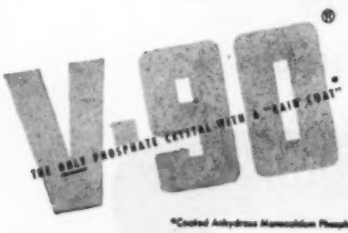
12 YEARS

MAKES SENSE

a sensible safety factor for your self-rising flour

Naturally you want your self-rising flour to keep well. You want it to bake even better than the quality of the flour warrants, if that's possible. And you want the oven products to have the natural, wholesome, *good flavor* you've milled into the flour itself.

This being the case, you'll be guided by Victor's 54 years of experience in developing leavening ingredients for self-rising flour. You'll pay special attention to V-90 . . . the *ideal* leavening with the *long* life. You'll look at V-90's record of 12 years of making *any* self-rising flour keep longer and bake better. You'll remember that V-90 is the ideal leavening that improves the flavor of oven products. Then you'll find that practically all of the self-rising flour made, contains V-90 and you'll say . . . "V-90 is for me!" *That* makes sense!



*Coated Anhydrous Monocalcium Phosphate

VICTOR CHEMICAL WORKS
141 West Jackson Blvd. • Chicago 4, Illinois

Service When It Counts

Every so often, we all need assistance and need it badly, to furnish the answers to many of our problems. In the Flour and Cereal Industry, N-A's Flour Service Division has been supplying practical, down-to-earth solutions to many of the industry's problems for over 25 years. Using its broad background of experience in the manufacture of time-tested products for maturing, bleaching, and enriching, the N-A Flour Service Division is ready to put its nation-wide service organization and extensive laboratory and research facilities at your service. They will work closely with your own staff and consultants to produce a concrete, individual solution to fit your particular needs.

For more details, call your nearest N-A Representative today and find out how this service can work profitably for you.

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